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A SERIES OF TEXTBOOKS FOR PERSONS ENGAGED IN ENGINEERING PROFESSIONS, TRADES, AND VOCATIONAL OCCUPATIONS
OR FOR THOSE WHO DESIRE INFORMATION CONCERNING THEM. FULLY ILLUSTRATED

MANUFACTURE OF PAPER
MANUFACTURE OF SUGAR
MANUFACTURE OF BEET SUGAR

SCRANTON
INTERNATIONAL TEXTBOOK COMPANY

1924

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PREFACE

The volumes of the International Library of Technology are made up of Instruction Papers, or Sections, comprising the various courses of instruction for students of the International Correspondence Schools. The original manuscripts are prepared by persons thoroughly qualified both technically and by experience to write with authority, and in many cases they are regularly employed elsewhere in practical work as experts. The manuscripts are then carefully edited to make them suitable for correspondence instruction. The Instruction Papers are written clearly and in the simplest language possible, so as to make them readily understood by all students. Necessary technical expressions are clearly explained when introduced.

The great majority of our students wish to prepare themselves for advancement in their vocations or to qualify for more congenial occupations. Usually they are employed and able to devote only a few hours a day to study. Therefore every effort must be made to give them practical and accurate information in clear and concise form and to make this information include all of the essentials but none of the non-essentials. To make the text clear, illustrations are used freely. These illustrations are especially made by our own Illustrating Department in order to adapt them fully to the requirements of the text.

In the table of contents that immediately follows are given the titles of the Sections included in this volume, and under each title are listed the main topics discussed.

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MANUFACTURE OF PAPER

(PART 1)

MATERIALS FOR MAKING PAPER

INTRODUCTION

1. Historical.—The exact time when paper was first used is not recorded. The substance which the Egyptians made as early as 2400 B. C. from the papyrus (from which the word *paper* is derived) was not a true paper. The Chinese made paper from cotton in the 2d century B. C., and are probably the true inventors of paper. The process employed in making cotton paper was unknown to the rest of the world, however, until the Arabs made it in 704 A. D.

The manufacture of paper was begun in Spain in the 11th century, and paper was made in France as early as 1189, but the industry made little progress in the latter country. The first paper manufactory was established in Germany in 1390 but the industry did not appear in England until 1490. In America, the first paper mill was established in 1690 at Germantown, near Philadelphia, by William Rittenhouse. Since that time the industry has advanced rapidly, and at present there are more than 2,000 pulp and paper mills in the United States and Canada.

2. Influence of Chemistry.—The rapid progress of the paper industry is largely due to the invention of the paper machine by the Fourdrinier brothers, about 1803, and to the application of chemistry. The influence that chemistry has

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had on the advancement of the paper industry cannot be overrated.

A discovery made by a chemist led to the utilization of wood as a raw material and the isolation of the large group of celluloses that enter into different grades of paper. The development of the Solvay and electrolytic processes for the manufacture of soda, the discovery of the bleaching action of chlorine, and the subsequent manufacture of bleaching powder, together with the discovery and application of coal-tar dyes, were great steps in the advancement of this industry. It is now necessary for a good paper maker to have a considerable knowledge of chemistry, in order that he may understand what actually occurs in his processes and be able to regulate conditions in order to obtain the best results. It is the aim to give a practical idea of the paper industry, and at the same time to show the chemical change that takes place and to set forth methods for testing and keeping a check on the ingredients used.

3. Usable Materials for Making Paper.—Paper is now made from the fiber of many varieties of plants, and from materials (rags, for example) originally made from vegetable fibers. In a treatise of this kind it is impossible to enumerate and describe all the different materials that could be used. Almost any form of vegetable matter could, with proper treatment, be reduced to pulp which could be used as a basis for paper. The determining factors as to whether such a line of manufacture is attractive or not are the value of the product and the cost of manufacture.

Scientific research will indicate whether a usable paper can be made from a given material, but will not yield an answer to the problem as to whether it can be made profitably. The locality has a considerable bearing on this. There are places where, on account of the distance of sources of supply of the usual raw materials, extraordinary materials can be profitably used to supply the local demand—for example, the use of waste sugar cane for paper in the Hawaiian Islands. A new paper material has small chance of success in most localities

unless it will give an equal amount of as good a quality of paper as the present materials, and offer at least as cheap a process of manufacture.

CELLULOSE

4. Cellulose.—Wood, straw, and most other vegetable structures are composed of innumerable fibers, so small that they can be seen only with the aid of a magnifying glass, and cemented together with other non-fibrous gummy or resinous materials, usually alluded to as *inter-cellular matter*. The fibers are composed of cellulose, and are of value for forming paper. The inter-cellular matter is of no value for forming paper. The aim of all paper-making processes is to wash or dissolve out all inter-cellular matter, leaving the pure fibers of cellulose in the form of pulp, which can be made into paper by mechanical treatment.

Cellulose is a definite chemical compound, with the empirical formula $C_6H_{10}O_5$, but of unknown molecular weight. It is of vegetable origin, and always exists in an organized condition, that is, in fibers of definite form. Chemically, all the different forms of cellulose are identical, but physically they are very different, depending on the kind of plant organism from which they have been prepared. Pure cellulose is perfectly white.

Cellulose enters into a great many chemical combinations. It can be oxidized, nitrated, hydrated, etc. These reactions however, have but little bearing on the ordinary problems of paper making. It is, on the other hand, important to note that cellulose is insoluble in all ordinary liquids, without undergoing chemical change. The only chemical reagent that will dissolve cellulose is *Schweitzer's reagent*, which consists of copper hydroxide dissolved in ammonia. This reaction is of practical importance in making artificial silk, fiber products, and also in the numerous tests for cellulose.

5. Cellulose Content of Different Materials.—Cotton fiber, which is more nearly pure cellulose than any other naturally

occurring substance, contains about 90 per cent. cellulose; linen contains from 70 to 80 per cent., depending on the quality of the flax from which it is made; spruce wood contains from 51 to 56 per cent., depending on the variety and the age of the tree; jute, from 60 to 64 per cent.; various straws, from 48 to 53 per cent.; esparto grass, from 46 to 59 per cent.; and manila hemp, about 50 per cent.

Such figures are the result of laboratory experiments and do not bear any definite relation to the amount of pulp that could be produced from such substances under practical working conditions.

Table I gives the yields of paper from various materials on a practical scale.

TABLE I
YIELDS OF PAPER ON MANUFACTURING SCALE

MATERIAL	PER CENT. PAPER
Rags	70 to 80
Esparto	40 to 45
Straw	40 to 50
Wood (by sulphite process).....	40 to 50
Waste fibers, waste paper, bagging, etc.....	75 to 90
Bamboo	40
Jute	50

It should be remembered that in general the percentage yield varies with the quality of the paper being produced, and usually the higher the quality of the paper, the lower the percentage yield, owing to the greater severity of the processes to which the raw material is submitted.

6. Fibers Used in Paper Making.—The fibers used in paper making can be roughly divided into four classes: (1) Seed hairs, of which cotton is the only example; (2) bast fibers, such as linen, jute, manila, etc., *bast* being a botanical term for the inner layer of the bark of trees, or the outer tough integument of plants which have not true bark; (3) fibers from whole cells or leaves, such as straw and esparto; (4) fibers derived from wood.

The first class of fibers finds its way into paper making chiefly as rags; the second as rags and jute materials. The third class is used in straw board, and also in papers made from esparto. The fourth class of fibers is most important in America, being the raw material of most of our paper—news-print, magazine and book paper, bag and wrapping paper, and all but the finest writing papers.

Before passing on to the main portion of this treatise, the manufacture of pulp and paper from wood, the various raw materials of paper and the process of making pulp from rags, from esparto and from straw and jute will be described.

7. Principal Materials.—The materials brought into most general use in the manufacture of paper are cotton, linen, straw, esparto, wood from various kind of trees, and jute, all of which, under proper treatment, will produce good fibrous stock that can be readily used.

8. Cotton and Linen.—For some time cotton and linen in the form of rags were the only materials used in the manufacture of paper by machinery, and they are still extensively used in the manufacture of the better class of writing paper. Cotton is the purest form of cellulose and possesses the quality of durability, being remarkable for its resistance to the action of caustic soda. Linen is cellulose isolated from flax by treatment with caustic soda. Müller gives the following analyses of cotton and linen:

COTTON		LINEN	
	PER CENT.		PER CENT.
Water	7.00	Water	8.60
Cellulose	91.35	Cellulose	81.99
Fat40	Fat and wax.....	2.37
Aqueous extract50	Aqueous extract	3.62
Ash12	Pectous substance....	2.72
Cuticular substance ..	.63	Ash70
Total	100.00	Total	100.00

9. Straw and Esparto Grass.—In the manufacture of cheap grades of paper, straw and esparto grass are used extensively. Straw is not so valuable as esparto, for the reason that it has to undergo a more severe treatment, which reduces the yield of the pulp greatly. Straw is used in America chiefly for the product known as strawboard. Esparto is used in England much more than in America and is often alluded to as *grass* by British paper makers. The following are analyses of straw and esparto according to Müller:

ESPARTO (SPANISH)		STRAW (RYE)	
	PER CENT.		PER CENT.
Cellulose	48.25	Cellulose	47.69
Fat and wax	2.07	Fat and wax	1.39
Aqueous extract	10.19	Aqueous extract	9.05
Pectous substance	26.39	Non-cellulose	26.75
Water	9.38	Water	11.92
Ash	3.72	Ash	3.20
Total	100.00	Total	100.00

10. Wood.—Owing to the large amount of wood available, the fiber of this material now holds the most important position in the manufacture of paper. A great variety of wood is used for this purpose, and the fiber of the wood is freed from the intercellular substances by either the sulphite, soda, or sulphate process, all three of which will be described later. For cheap grades of paper, the wood is ground up and pulped without removing the intercellular substances. The chief woods used, together with the yield of pulp per cord and the process by which the fiber is isolated, are given in Table II.

11. Spruce is by far the most important of all species of wood for both mechanical and chemical pulps. From a chemical standpoint, it is desirable because it contains a maximum percentage of cellulose. The paper maker prefers it because the fibers are longer, more flexible, and stronger than those of most other woods. Moreover, comparatively

abundant supplies are available throughout the United States and Canada. Hemlock probably ranks second in importance. For most purposes it is inferior to spruce, but hemlock sulphite pulp is excellent for high water-finish papers, such as the wrapping paper commonly used in drug stores. Next to spruce and hemlock in importance comes balsam, but balsam pulp is very inferior to spruce pulp, even when made with great care. Poplar and aspen are largely used for making certain sorts of pulp. A great deal of soda poplar is used in soft, bulky papers, and mixed with other pulp in book and magazine papers. All other woods are of far less importance than the five mentioned.

TABLE II

YIELD OF PAPER PULP FROM VARIOUS KINDS OF WOOD

Wood Used	Yield of Pulp Per Cord, in Pounds			
	Soda Process	Sulphite Process	Sulphate Process	Mechanical Process
Poplar	1,000 to 1,500			1,400 to 2,000
Spruce		900 to 1,300	900 to 1,100	1,600 to 2,300
White pine			900 to 1,000	1,600 to 2,000
Basswood	900 to 1,000			
Hemlock		700 to 1,000	700 to 1,000	
Spruce and poplar	1,200	900 to 1,000		1,600 to 2,000
Spruce, poplar, and pine				1,000 to 1,200
Spruce and hemlock		1,100		

12. Jute.—The material known as jute, which is cultivated in Bengal, consists of the bast, or fibrous inner bark, of *Corchorus*. It is used to a great extent in the manufacture of paper. This fiber cannot be bleached white and jute is therefore used mostly in the manufacture of strong wrapping paper, etc., the strength of which is of more importance than the appearance. The composition of jute is shown by the following analysis:

	PER CENT.
Water	10.92
Aqueous extract	1.43
Cellulose	62.96
Fat and wax41
Non-cellulose	23.53
Ash75
Total	100.00

13. Miscellaneous Fibers.—Flax, hemp waste, manila, cotton linters, bagasse (the refuse from cane-sugar factories), waste paper, bamboo, and the inner bark of the paper mulberry are also used to some extent in the manufacture of paper. Woolen rags are used to a moderate extent, but as it is almost impossible to bleach them, they are only mixed with other materials in the manufacture of coarse papers and wrappers.

MANUFACTURE OF PULP

14. Before wood, rags, and other materials can be made into paper, they must undergo some treatment by which the fibers are separated from one another, reduced to a certain degree of fineness, and, for the better qualities of paper, all resinous and other foreign matter removed. This is accomplished in various ways, the product being known as *pulp*. The methods of treatment of different substances in the manufacture of pulp will here be discussed separately.

RAG PULP

15. Sorting of Rags.—Rags as they are received at the factory are unfit for immediate use. They must first undergo a preliminary treatment of sorting, cutting, and dusting before they are ready to be made into pulp. The rags are taken from the bales and sorted as to condition of wear, color, the nature of the fiber, that is, whether linen, cotton, etc., the better qualities being used in the preparation of pulp for the better

class of paper. At this point of the process, buttons and other materials mixed with the rags are removed

The usual grades of rags are: (1) New white linen cuttings; (2) new white cotton cuttings from textile factories; (3) fine whites (domestic rags); (4) seconds (grade next to *finest*); (5) thirds (dirty, well-used rags); (6) colored rags (all grades of colors); (7) canvas; (8) manila and hemp rope; and (9) bags and jute.

16. Cutting and Cleaning of Rags.—Many different types of machinery are used to cut rags. In the older mills the *guillotine cutter* is sometimes used. It is simply a descending knife that cuts off the rags pushed beneath it. A more improved type consists of an iron table bolted on the top of a cast-iron stand. This table supports the bearings of two driving shafts. To one of these shafts, driven by a tight and loose pulley, a cutter head is attached, carrying a number of fly knives. At the other end of this shaft is a flywheel to relieve the shock when cutting. The fly knives cut against a bed knife fastened to the table. A separate belt, on a small pulley to the right of the operator, propels a series of gears which drive a drum studded with projections. This drum feeds the rags to the knives. It is carried on a curved frame, which may be lifted should the rags clog the machine. The length of the rag cut is controlled by the difference in speed given to the two belts. Such machinery will cut from 1,000 pounds to 1 ton of rags per hour.

17. After cutting, the rags have to be *dusted*, that is, they have to have the loosely attached dirt removed as far as this is possible. The machines used in the operation are variously called *dusters*, *willows*, and *thrashers*. The general principle is a revolving cylinder or cone, fitted internally with flanges or spikes, which permit the rags to be opened up and beaten. The dust usually falls out through a perforated base. The nature of this equipment varies according to the kind of rags treated.

The entire operation of preparing rags for boiling is a dangerous one to the health of the workers, owing to the

disease germs carried by the dirt in many kinds of rags, as well as the irritation of the dust. Consequently, all such equipment should be enclosed as far as possible and good forced-draft ventilation provided, as well as plenty of air and sunlight.

18. Boiling Rags.—Rags are usually boiled in vessels of steel plate, which may be of either spherical or cylindrical

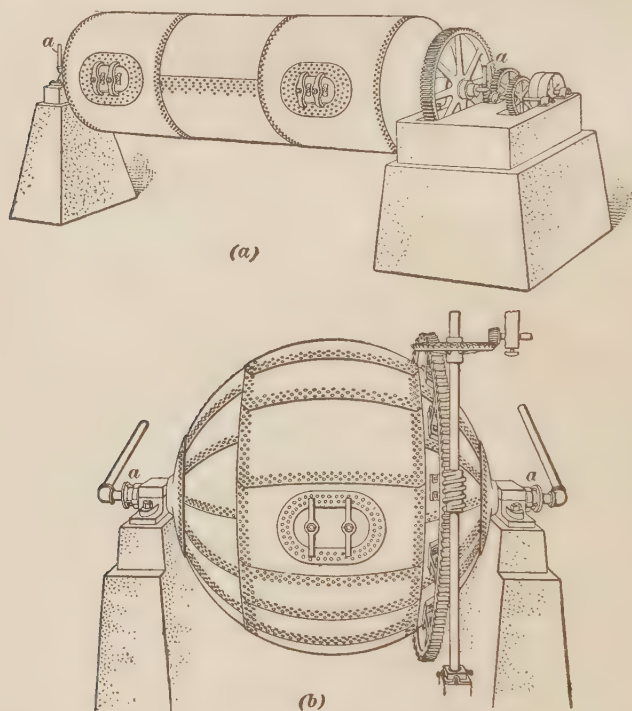


FIG. 1

shape, as illustrated in Fig. 1 (a) and (b). The object of the boiling is to remove the grease and dirt by means of the high temperature, the agitation, and the chemicals used; and to bring the rags into such a condition that the impurities can easily be removed by washing, leaving fiber suitable for paper.

Cylindrical boilers are now most usual, ranging from 6 feet in diameter by 10 feet long (capacity 270 cubic feet) to 8 by

24 feet (capacity 995 cubic feet) and rotating from one-third of a revolution to three revolutions per minute. The trunnions *a* are hollow, for the admission of steam. In some cases ribs, or flanges, are arranged around the inside to prevent the stock from packing against the sides. The internal pressure under operation ranges from 60 to 120 pounds per square inch.

19. Caustic soda or milk of lime is added to the rags and water in the boiler. For fine papers, caustic soda is chiefly used, and for coarser papers milk of lime. Mixtures of the two are also used. About 15 pounds of lime per 100 pounds of rags is usually sufficient, and from 5 to 10 pounds of caustic soda per 100 pounds of rags.

The usual practice is to fill the boiler about two-thirds full with rags and the chemical solution and run from 20 minutes to $\frac{1}{2}$ hour before steam is admitted. Steam is then turned on and the boiling continued for from 2 to 6 hours, depending on the nature of the stock and the strength of the chemical used. The lower the grade of the rags, the stronger must be the chemicals used, the longer the boiling period and the higher the temperature and pressure.

20. Various practices prevail with regard to emptying the boiler at the end of the operation. Some operators relieve all the pressure before removing the stock; others blow off under pressure. It is claimed for the latter procedure that it gets rid of any dirt remaining in the stock, but it is undoubtedly hard on the fiber.

After the rags are boiled, they are allowed to drain on draining floors, which are simply perforated floors of wood or metal. Sometimes the stock is allowed to drain for several days to soften it.

21. Washing and Breaking.—After the rags are drained, they are taken to a rag engine, such as that illustrated in Figs. 2 and 3. In this engine, the rags undergo a thorough washing, and the breaking up of the fibrous matter is accomplished.

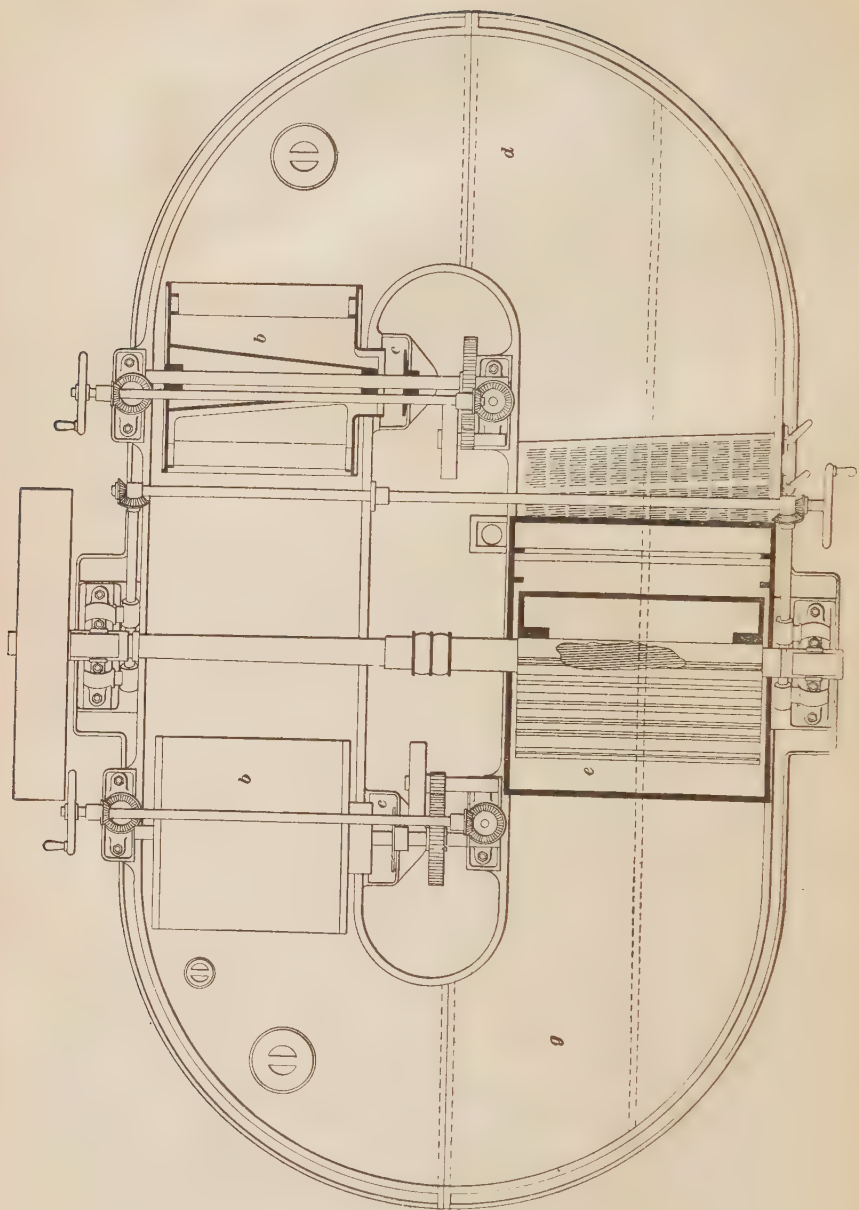


FIG. 2

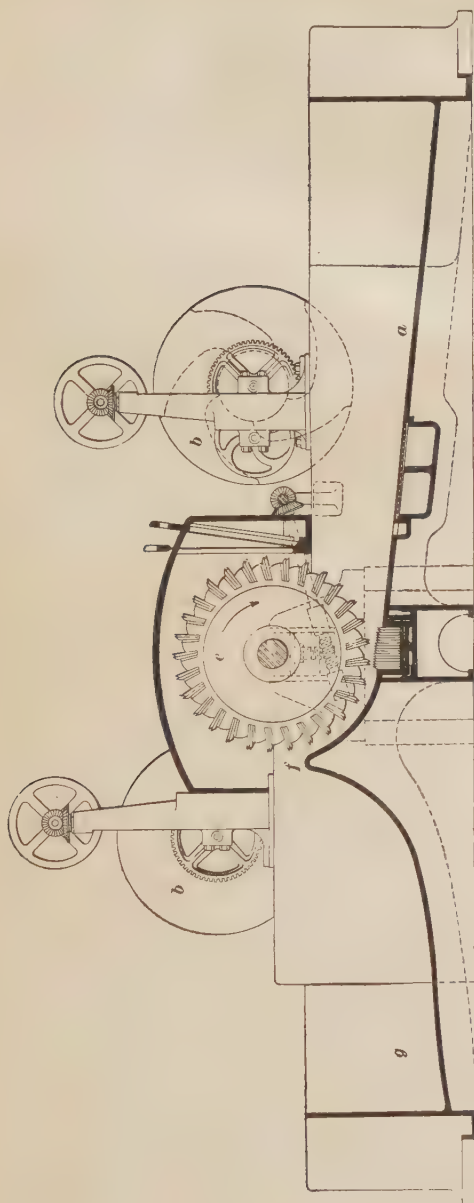


FIG. 3

22. Rag Engine.—The ordinary rag engine, which is illustrated in Figs. 2 and 3, consists of an oval tank divided in the center longitudinally by a partition called the *midfeather*. In one side is situated a roll *e* bearing knives around its circumference. Directly under this roll, in the bedplate, are knives set with their cutting edges in the opposite direction. The distance between the knives on the roll and those in the bedplate can be varied at will so as to regulate the degree of fineness to which the rags are cut. The floor *a*, Fig. 3, of this side of the tank is inclined and has a raised portion *f* that keeps the rags well under the roll. On the opposite side of the tank

are placed the drum washers *b*. The washers are either cylindrical or octagonal drums of wire cloth, the ends of which are made of wood. This construction permits the water to flow through the washers and to pass out but the pulp is held back by the wire cloth.

23. When washing, the tank is partly filled with water and the boiled rags are fed in at *d*, Fig. 2. The roll *e*, revolving in the direction shown by the arrow carries the rags between the knives of the roll and those on the bedplate, where they are cut. The cut rags pass over the raised portion *f* into the part *g*, from where they pass on to the drum washers, and the dirty water passes out. The supply of fresh water is such that a constant level is maintained. The washing is continued until the wash water passes out clear. The supply of water is then shut off and the washer kept in motion, being lowered as the level falls, until completely drained. The bleaching is sometimes done in the washer itself, but generally in what are called *potchers*. The bleaching of rags will be taken up later.

ESPARTO PULP

24. Esparto Grass.—The esparto grass is received in bales and undergoes a treatment somewhat similar to rags. The bales are undone, and the small bundles are put into the hopper of a machine called a *willow*, consisting of several revolving cylinders with projecting teeth that agitate the grass violently and thereby remove the dirt, which falls below the machine, where it is removed by means of a fan. The esparto is then taken to the boilers, which are usually upright and hold from 2 to 4 tons of grass each. The grass is boiled in caustic soda, the strength of which is about $9\frac{1}{2}^{\circ}$ Baumé, under a pressure of about 20 pounds, the time required for boiling being about 3 hours at full pressure. The pressure is then blown off, and, after running off the spent liquor, the grass is again boiled with water for about $\frac{1}{2}$ hour, after which it is well drained and removed to the washing engine by means of a door at the bottom of the boiler.

The amount of caustic soda used, the pressure at which the esparto is boiled, and the time required, all vary with the character of the boiler used and the sort of grass treated. The range is from 14 to 16 pounds of 70 per cent. caustic soda for each 100 pounds of grass; from 30 to 40 pounds of pressure; and from $2\frac{1}{2}$ to 3 hours of boiling. In this boiling action, the fatty and resinous bodies are converted into soluble soaps, the silica of the grass is partly dissolved as silicate of soda, and the complex cellulose is split up into cellulose and soluble derivatives.

25. Washing of Esparto Pulp.—The washing of esparto pulp is first carried on in engines similar to the rag engine; after this the drum is allowed to run until there is room for the bleach liquor, when it is added, and the bleaching conducted in this engine. As quite a large proportion of the fiber is lost by using the washer just mentioned, many paper makers object to this machine. Very good results have been obtained by using a series of tanks, arranged one above the other. In this method fresh water enters the bottom of the upper tank, and, overflowing from the top of this tank, enters the bottom of the next lower tank, and so on, the motion of the water being very slow, so that the finer particles are not carried away.

The yield of esparto fiber depends, of course, as in the case of wood, on the strength of the liquors used, the time of cooking, and the quality of the grass.

PULP FROM STRAW, JUTE, AND OTHER MATERIALS

26. Straw requires a more severe treatment than esparto or rags, as the knots in the straw must be reduced, and this, of course, necessitates the use of an excessive amount of soda. By this treatment, the finer fibers are liable to be carried away with the wash water, and the yield has been as much as 10 per cent. less than that of esparto. Wheat, oat, rye, and barley straws are generally used for making straw pulp, wheat and oat straws forming the bulk of the material used in the United States. From 10 to 20 pounds of caustic soda for each 100 pounds of straw is required to boil the straw thoroughly.

27. The following is a description of the usual process for making paper pulp from straw. For making strawboard pulp, milk of lime is used instead of caustic soda. The straw is first cut into short lengths by means of a cutter similar to the rag cutter. The cut straw is then conveyed through a wire cylinder to remove any dust, after which it is sent to the boiler. The boiler is usually of a cylindrical, rotary form, although some paper makers prefer to use a stationary boiler. On account of the straw being so bulky, the boiler is first partly filled with caustic liquor. The straw is added until the full amount has been put in, then the balance of the liquor is run in, and the head of the boiler is screwed into place. The straw is cooked from $3\frac{1}{2}$ to 8 hours at a pressure of from 29 to 40 pounds. The strength of the liquor varies from 6° to $10\frac{1}{2}^{\circ}$ Baumé. When the boiling is complete, the boiler is allowed to cool and the charge is run through a pipe to the drainer tanks. These are large tanks provided with perforated bottoms that permit the liquor to be drained out and the stock washed to some extent. The straw pulp is then usually transferred to a breaking engine where the washing is completed, after which it is pumped over sand traps, which are long, shallow trays containing boards stretched from side to side, sloping at an angle, and nailed to the bottom of the trays. The dilute pulp flows over the trays, leaving the heavy particles, knots, etc., behind the sloping boards. It then passes through steamers and is conveyed to the bleachers.

28. A low-grade pulp, suitable for the manufacture of the coarser grades of paper, such as building paper and coarse wrapping paper, is made from such materials as jute, flax, manila, and hemp waste. The materials are prepared by cutting and boiling by processes very similar to those used in the treatment of esparto and straw. Jute is usually boiled with milk of lime instead of caustic soda.

The materials just mentioned do not require such careful treatment as is necessary in the manufacture of the better grades of pulp.

MAKING WOOD PULP

CLASSIFICATION OF METHODS

29. By far the greater quantity of paper used at present is made from wood pulp. Pulp is made from wood by two distinct classes of methods. The first class includes the *mechanical process*, which results in mechanical wood pulp, or ground wood. The second class includes the various chemical methods, which produce chemical pulp or cellulose; and these chemical methods are three in number: (1) the sulphite process; (2) the soda process; (3) the sulphate, or kraft process

MECHANICAL PROCESS

30. Mechanical Pulp.—The pulp produced by the mechanical process is inferior to that produced by the chemical processes and, consequently, it is only used in making those kinds of paper where high quality is not demanded and price is the chief consideration, for example, in newsprint, or else in judicious combination with chemical pulp, as in the manufacture of certain bags and wrapping papers. The inferiority of paper containing mechanical pulp is due to the shortness and weakness of the fibers, together with the fact that such paper will quickly deteriorate and turn yellow owing to the action of the atmosphere on the lignins contained in the wood pulp.

Mechanical pulp is, however, much cheaper to make than any other form of paper pulp. In the first place, only about 2 per cent. of the raw material is lost, as compared to 50 per cent. or more in the chemical processes. Secondly, no chemicals are required. Finally, the equipment necessary for making mechanical pulp is much cheaper, both as regards first cost and maintenance than that required for making chemical pulp.

31. Semichemical Pulp.—Semichemical pulp is a variety of mechanical pulp in the manufacture of which the wood is steamed and softened before being ground. In this way

certain characteristics are brought out that make the pulp more suitable for some finished products, and stronger and more flexible than that produced by the ordinary mechanical process. Owing to the action of the steam on the wood, the fibers are colored brown as a result of oxidation, and this limits the application of this kind of pulp to uses such as the making of brown boards and wrapping paper, where this color is not objectionable.

32. Power for Ground-Wood Mills.—Ground-wood mills are ordinarily located where there is plenty of available water-power; otherwise steam or electric power is used. Up to within a few years ago, all motive power for the operation of pulp grinders, screens, presses, etc., was supplied by waterwheels, but today the electric motor-driven, ground-wood mill is finding an increased field, due to the fact that uniform water power is not always available.

33. Effect of Wood on Pulp Quality.—The quality of mechanical pulp is determined largely by the texture of the wood, the age, and the seasoning. Wood for grinding must be sound; that is to say, the interior must not be reddened by disease, and neither blue nor black, owing to the fact that the felled trees are exposed too long to the damp air.

The best wood is that which has been preserved on a dry and very airy spot, stacked consecutively so as to allow active circulation of air, and used within 6 to 12 months after cutting. The fresher and greener the wood the more easily will it grind, the yield will be higher and less power will be consumed. It is more difficult to secure a long, free stock from wood that is dried and has been seasoned, than from good fresh material.

34. Grinders.—The type of grinder universally used is illustrated in Fig. 4. It consists of a large grindstone, usually about 54 inches in diameter, and wide enough to accommodate 16-inch or 24-inch blocks. There are some grindstones in operation that are 54 inches wide and that accommodate 4-foot wood. The grindstone is mounted on a shaft and revolves

inside an iron casing *a* which usually has three compartments *b*. At the upper extremity of each of these compartments is a hydraulic cylinder *c* fitted with a piston, and as the wood is fed into the compartment, the piston forces it against the revolving stone. The pressure is furnished by either a centrifugal pump or a duplex or triplex plunger pump. The pump is usually driven from the grinder spindle. The friction between the stone and the wood causes intense heat,

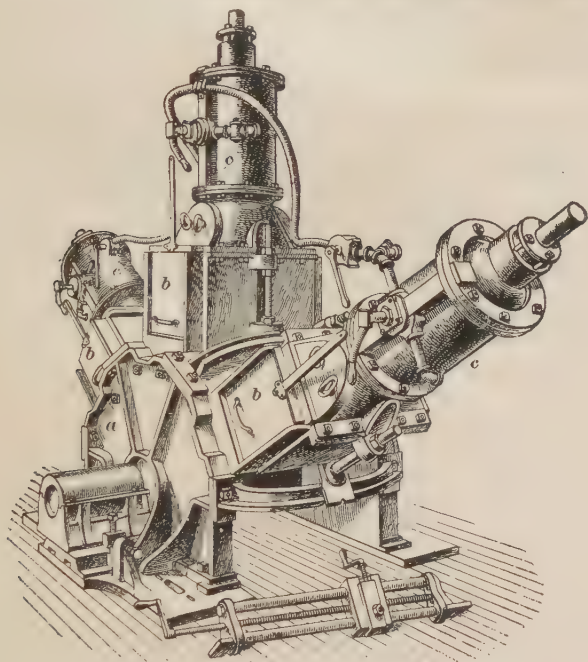


FIG. 4

which necessitates the use of a large stream of water, the object of which is to clear the stone and also reduce the temperature of the operation. Failure to keep the temperature sufficiently low causes the stone to become glazed and the stock is burned lifeless and short. The capacity of the grinder depends on the nature of the wood and the power available, but it usually runs from 2 to 7 tons per 24 hours and requires approximately 200 to 600 horsepower per grinder.

35. Screening.—As the ground-wood pulp reaches the grinder pit, it falls to the sliver screens, a series of iron plates perforated with $\frac{3}{8}$ -inch or $\frac{1}{2}$ -inch holes, that remove the larger bull slivers, knots, etc., which are carried off from the surface of the screens by a drag conveyer, dumping the bull slivers in a receptacle at the top of its course. The screen and conveyer are constantly showered with water to wash off any fibers adhering to the large slivers.

The stock freed from these large slivers is pumped to a centrifugal or diaphragm pump screen, which removes the last traces of slivers and coarse stock. This stock pump is either a slow-speed fan pump or a plunger-type pump. An 8-inch fan pump will handle the stock from four three-pocket grinders under ordinary conditions and will pump the stock to the screens with approximately 20 horsepower. This figure is for a stock line 8 inches in diameter and approximately 150 feet in length. Through these screens only the finest fibers are permitted to pass, the rejected coarser particles being passed through a machine for reducing and refining them, after which they are again passed over screens.

36. Pulp Thickener.—At this point in the process the stock contains about one-half of 1 per cent. of pulp, as a large amount of water is required for disintegrating and washing and screening—approximately 150,000 gallons of water per ton of air-dry product. The removal of a certain percentage of water from this very dilute mixture is ordinarily performed by means of a pulp thickener, which is a cylinder revolving in a vat of stock, the cylinder being fitted with a wire netting of about six meshes to the inch, which supports a finer wire cloth. The top of the cylinder is above the contents of the vat and as it revolves the water passes through to the inside and leaves a thin layer of pulp on the outside. At this point the machine resolves itself into either one of two types, depending on whether deckered stock or pressed stock is to be made. If stock is to be used in the mill direct, it can be scraped off from the top of the cylinder with a doctor and then pumped to the beater as required. This operation is

termed *deckering*, while the machine, consisting of vat, revolving cylinder, and doctor for removing the stock, is termed a *decker*. The other type is that of the wet machine or press.

37. The **wet machine**, illustrated in Fig. 5, is used for that portion of the stock which cannot be deckered and used as deckered stock. It is practically a decker with an endless band of felt and press rolls added. There is a vat *a* in which a roll *b* covered with wire cloth is revolving. The felt goes round the couch roll *c* so that it comes in direct contact with the film of pulp that is picked up by the cylinder. This film

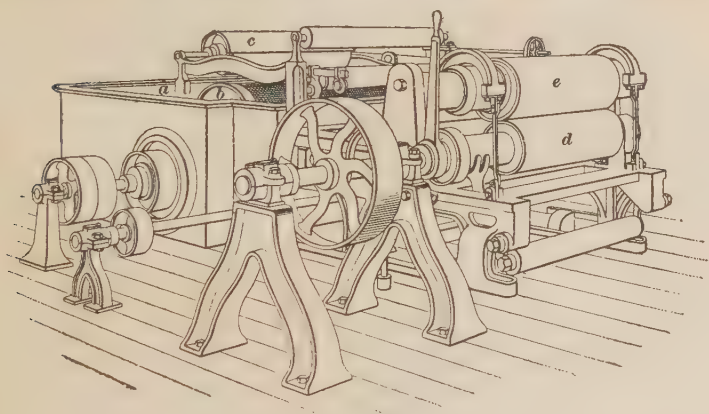


FIG. 5

is carried forwards on the felt between the press rolls *d* and *e* and allowed to wind around the top press roll *e*. When it becomes thick enough it is cut off with a wooden pin. The size of the sheet thus made is governed by the circumference and width of the top press roll. This sheet is deposited on a folding table in the front of the press roll and folded lengthwise twice and crosswise three times, making a sheet of pulp about 18 inches by 24 inches. Such sheets are called *laps*.

38. The wet machine is driven through the bottom press roll *d*, Fig. 5, the felt acting as a belt, driving the cylinder and the carrying rolls.

Pressure is applied to the top press roll by means of a system of compound levers with weights, or by springs, or occasionally by hydraulic pressure. Hydraulic pressure is used chiefly by mills that prepare pulp for shipment to a distance, in which case it is desired to eliminate all the water possible. In such cases the pressing on the wet machine is frequently followed by further pressing in a specially designed hydraulic press.

The ordinary wet machine, equipped with levers or springs, can produce a pulp approximately 40 per cent. dry. The packings on the ends of the cylinders should be kept perfectly tight so that the stock does not leak through and go into the white, or waste, water.

Should the pulp suddenly become full of sheaves and dirt the trouble can usually be traced to the screen plates of the centrifugal screens, and it will be found, very likely, that a piece has broken right out of one of these, or that they have been cracked. Another cause of dirty pulp suddenly appearing is a dirty vat. Pulp frequently shows dirt for this reason when a wet machine is started after having been shut down.

39. Color and Quality of Ground Wood.—The color of ground spruce wood is yellow, but of a lighter shade than the original wood. To bleach ground spruce to pure white is practically impossible. In the manufacture of paper from ground-wood pulp, the yellow color is usually neutralized by means of aniline blue. This produces a fairly white effect, but it is neither so bright nor so white as can be obtained from bleached stock.

Good ground wood should be of a rich, creamy color. This indicates that the grinders are not allowed to run too hot, and that the pulp is not burned. The flow of the pulp over the grinder dams should reveal to the experienced eye the texture of the pulp. Coarse pulp will fill up to the top of the dam and break over it in chunks instead of flowing over smoothly.

40. A piece of blue glass set in a small wooden frame is very useful for testing ground wood. A sample of the ground wood is taken and diluted with water. If the frame is now

held against the light, the fibers being white and light blue, the size, nature, and uniformity of the fibers can be seen very clearly. If chips, sheaves, slivers, or unseparated bundles of fibers are present, they will be revealed.

Another useful device is a long, sharp-pointed stick for detecting the presence of holes and uneven spots on the surface of the stone. If such a stick is held against the surface of the stone as it revolves, and gradually moved across the surface, any irregularities in the surface will reveal themselves by making the stick vibrate. The sense of touch will soon learn to detect even very slight defects in the surface of the stone. This device obviates the necessity of shutting down the grinders to find out whether the stones need burring.

41. Burring.—Closely allied in importance to the choice of raw materials is the question of the *burring*, or sharpening, the stones, as the surface of the pulp stones has an extremely important effect on both the quality and the quantity of pulp that the grinder will produce. Stones suitable for grinding pulp for coarse papers would not be suitable for making a light-weight sheet containing from 40 to 60 per cent. of ground wood. A coarse stone should be used for coarse pulp, while a stone with a finer grit should be used for fine pulp. Since stones vary in texture to a large extent, these natural variations must be regulated by proper burring of their surfaces so that this irregularity will not show up in the finished pulp.

The burring operation is performed by means of a steel burr placed against the revolving stone. There are many different types of burrs on the market, all of which differ slightly in pattern on the outside circular circumference. Tests have been made which show that if the stone is properly dressed the product will be good regardless of the type of burr.

When the grinder pressure and speed are held constant the effect of sharpening the pulp stone is to increase the production and produce a pulp of a coarser quality.

SULPHITE PROCESS

42. Nature of Sulphite Process.—The sulphite process is today the most important chemical process for making wood pulp. It was invented about 1867 by B. C. Tilghman, of Philadelphia, and improved by Eckman in Sweden, Mitscherlich in Germany, and Partington in America, as well as by many other workers. The process as it stands today is radically different from Tilghman's original discovery, but, nevertheless, it is derived directly from it.

In this process wood chips prepared properly are digested with an acid liquor at a high temperature and pressure. The acid liquor is chemically a solution of bisulphite of lime. It is frequently alluded to as *acid*, and the plant for making it in a pulp mill is called the *acid plant*.

This liquor has the power of dissolving and removing all the constituents of the wood chips except the cellulose, which, in impure form, constitutes the unbleached sulphite pulp. When bleached, this pulp is practically pure cellulose. The yield of bleached pulp will vary from 49 to 53 per cent. of the weight of the prepared wood.

43. Disadvantages of Sulphite Process.—The cost of making sulphite pulp is considerably more than that of mechanical pulp. In the first place, only about 50 per cent. of the raw material is retained in the finished product. Moreover, much labor and power must be spent on the preparation of the wood before it reaches the digesting process proper. It also involves the up-keep of a large chemical plant for making the acid liquor. Finally, the machinery is expensive, both as to cost and maintenance, the up-keep being high, as the acid nature of the process makes for rapid deterioration. The sulphite process requires about 1,300 to 1,500 pounds of coal per ton of pulp, 232 pounds of sulphur; and 300 pounds of limestone. However, the greater length and higher pliability and strength of the sulphite fibers, together with the freedom from deterioration, cause sulphite pulp to be used instead of mechanical pulp for all except the cheapest grades of paper, in spite of its higher cost.

44. Bisulphite Liquor or Acid.—Sulphur, when burned, units with the oxygen of the air to form sulphur dioxide, SO_2 , a gas that dissolves readily in water. There is always a tendency for sulphur dioxide to take up more oxygen, becoming oxidized to sulphuric acid. In fact, this is the way in which commercial sulphuric acid is manufactured. In burning sulphur for the manufacture of sulphite pulp, every precaution is taken to prevent the formation of sulphuric acid.

The liquid generally called *acid*, which is used in the sulphite digesters, consists of lime and magnesia bisulphites in solution, together with free sulphur dioxide in solution. The acid may also contain some sulphate and some monosulphate, but if the operation is properly conducted the amounts of these compounds will be negligible. Their presence is very undesirable.

45. Until recently, it has been the opinion of many that acid high in magnesia was to be preferred. The explanation of this has been that magnesium bisulphite is decomposed easier by steam in cooking than calcium bisulphite and that the sulphates of magnesia are soluble whereas the sulphates of calcium are insoluble. Possibly these facts had a bearing on the quality of pulp produced in former days when sulphite mills used acid low in free SO_2 and high in lime. Today, however, with excellent means of reclaiming the digester gases and obtaining cooking acid containing from 5 to 7 per cent. total SO_2 , the preference for magnesia over calcium has absolutely disappeared. Mills having changed from milk of lime to a straight calcium limestone have confirmed the above experience.

This acid is distinctly corrosive and has to be handled in equipment constructed of materials selected to resist its corrosive action. All pipes, connections, valves, pumps, etc., with which it comes in contact, must be of bronze, hard lead, or some other acid-resisting material.

46. Sulphur Burners.—Several different types of sulphur burners are in use. The older type, which has been largely replaced by more efficient devices, is the flat burner. This

is essentially a cast-iron retort, the body consisting of a single casting. The cross-section is semicircular or D-shaped. It is usually 8 feet long and 2 feet wide on the outside and the maximum height on the inside is about 18 inches. The burners are set in banks in a brick setting, a sufficient number being installed to provide the amount of sulphur dioxide required for making the volume of acid needed by the mill. The doors of the burners are thus ranged in a horizontal row at a convenient height from the floor so that the sulphur can be charged into them with shovels. When once ignited the sulphur maintains combustion if an adequate supply of air is admitted to the burner. The air supply is regulated by dampers in the doors and the regulation must be attended to by a careful, experienced man, because, if too much air is admitted, there is a tendency to form sulphuric acid, which forms sulphates in the sulphite acid, which are insoluble and cause trouble in the washing of the pulp. Moreover, if too much air is admitted, this excess air dilutes the gas produced in the burners and cuts down the efficiency of the tower system. On the other hand, an insufficient supply of air results in incomplete combustion of the sulphur and this causes sublimation, or deposit of unburned sulphur, throughout the system, which is troublesome and lowers the efficiency. The sulphur dioxide leaves the furnace through the pipe, which is bolted to the back of the furnace, or to the top of the arch near the back end.

47. A more efficient type of sulphur burner is the rotary burner developed by Tromblee and Paull, illustrated in Fig. 6. It consists of a cast-iron cylinder *a*, usually about 8 feet long and about 2 feet in diameter, mounted on brick settings *b*, and revolved slowly by means of gears *c*. At each end of the barrel is a cone *d*. The rear cone connects with the pipe *e*, which carries off the sulphur dioxide, and the front cone contains the damper by which the air supply is regulated. The revolution of the burner constantly keeps a fresh surface of sulphur exposed to the air, thus giving very efficient combustion. The sulphur is fed to the burner from hoppers *f*, directly

above the damper. A worm is generally used to facilitate the steady and equal feeding of sulphur to the burner. The gases are conveyed from the burner proper into a large combustion chamber, which is provided with dampers through which air can be admitted to complete the combustion of the sulphur. In any case, sulphur becomes sublimed, on account of insufficient air supply through the front damper of the burner and is caught in this chamber where combustion is completed. In this way it is prevented from getting into the cooler and into the acid system.

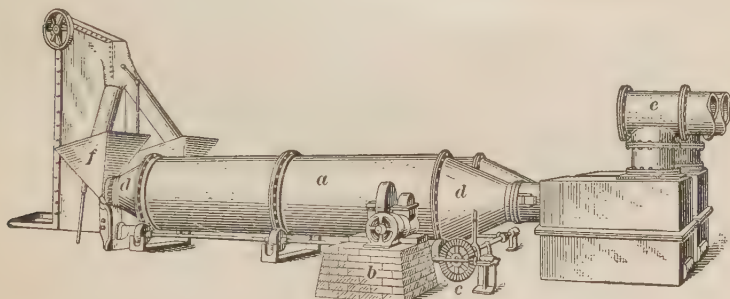
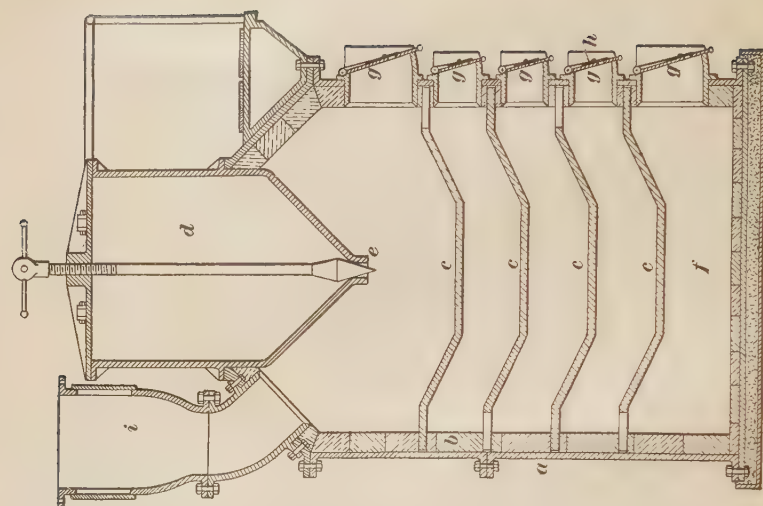
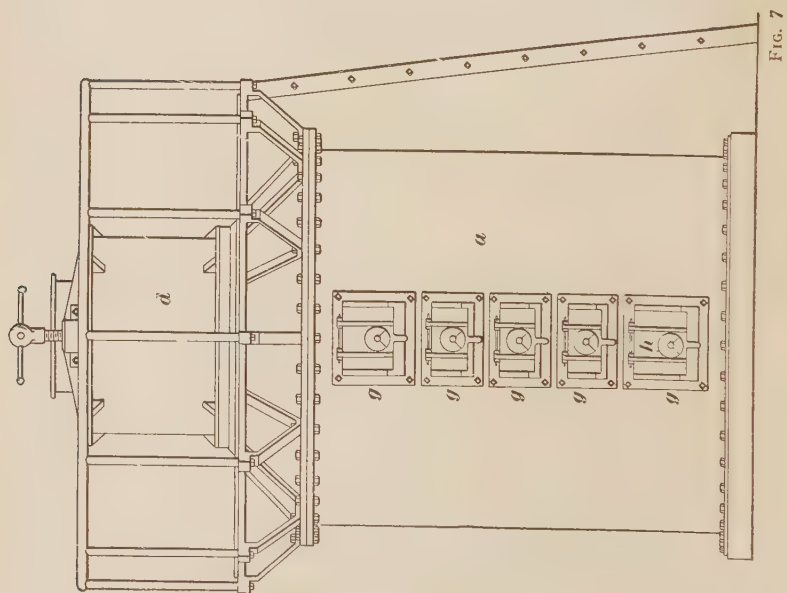


FIG. 6

48. Another type of sulphur burner is the Vesuvius burner, illustrated in Fig. 7. This consists of a cylindrical steel shell *a*, lined with firebrick *b*, containing a number of shallow trays *c*. The trays are so arranged that the molten sulphur will drip down from each tray to the one below, burning as it drips. The sulphur is placed in a melting pot *d* at the top of the burner. This melting pot has a needlepoint valve *e* in the bottom. A fire is lighted in the top tray and as soon as the sulphur begins to melt the needlepoint valve is opened and the sulphur drops onto the first tray and begins to burn. The heat soon melts all the sulphur in the pot and gradually the valve is opened wider and wider and the burning molten sulphur drips down from tray to tray until it enters the bottom chamber *f*, where the ashes and impurities collect. Each of the chambers is provided with a door *g* fitted with a damper *h* for the admission of air. From the burner the gases pass to a combustion chamber *i* wherein the sulphur



fumes undergo a final combustion. One of the advantages of this kind of burner is that it does not require any power to operate. Moreover, no hand firing is required. All ashes or impurities, called *slag*, are automatically carried to the bottom of the apparatus where they can be easily removed.

49. Coolers.—From the combustion chamber the sulphur gas is led to coolers, as it is necessary to cool the gas to about 25° C. before it enters the towers. If the air supply has not been carefully regulated sulphuric acid will be formed in the coolers. The volume of air required to burn 1 pound of sulphur to 15 per cent. SO_2 gas is 78 pounds, or 9 cubic feet. The burner should be so regulated that about 15 per cent. SO_2 gas is formed in the burners. The combustion chamber should be as large as possible and should contain baffles to collect the dust and sublimed sulphur. If the foregoing precautions have been observed and the gas is properly made, it will be cooled in the coolers without any considerable contamination with sulphuric acid and will also be free from sublimed sulphur.

50. There are numerous types of coolers in use. One type illustrated in Fig. 8, consists of two lead headers, an upper one and the lower one as at *a*. Into the headers is flanged a series of vertical lead pipes *b*. The whole is placed in a concrete tank *c* which is filled with rapidly circulating, cold water. The gas enters the lower header *a*, passes up through the cold vertical pipes *b*, and exits through the large horizontal pipe at *d*.

In order to obtain the maximum cooling effect of the water, it is made to enter the concrete tank at the upper header and leave at the lower header. The effect of this counter-current system is to bring the coldest water in contact with that end of the pipes through which the coldest gas is passing. In this way the gas is practically cooled to the temperature of the entering water. The warm water coming from the cooler is run into the sewer, or under certain conditions it may be made to serve a useful purpose. Thus, if the lead pipes are kept in a good state of repair, this water will be perfectly

pure and can be used for washing purposes or as boiler feed-water, thus utilizing the heat given up by the gas. This type

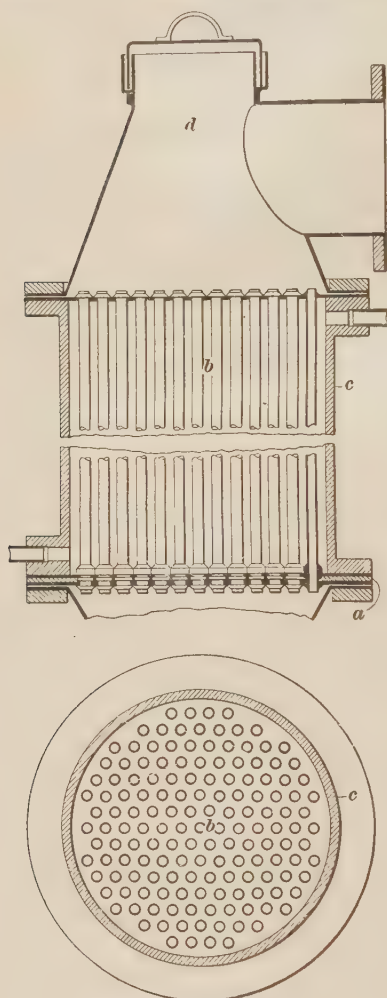


FIG. 8

of cooler is not only very efficient but is easily cleaned, as the top of the cooler is readily accessible. The cooler should be as near the combustion chamber as possible, so that the cooling of the gas will be rapid. Slow cooling tends to produce sulphuric acid.

51. Absorption Equipment.

—There are two chief classes of equipment in which the sulphur-dioxide gas is brought into contact with the lime and thus converted into acid for use in the digester. One form of equipment is generally known as the *milk-of-lime system*, in which the gas is brought into contact with water containing the lime in suspension. The second and more modern system of acid making is known as the *tower system*. In it the gas is brought into contact with limestone, not lime, in the presence of water. In the milk-of

lime system the gas first dissolves in the water and the solution immediately reacts with the lime to form monosulphite, which is quite insoluble and separates from the solution. The

formation of monosulphite goes on in the tank until all of the lime is precipitated in this form. As the addition of gas continues, the monosulphite gradually takes up more sulphur-dioxide gas, forming bisulphite, which, unlike the monosulphite, is readily soluble in water. Unfortunately, sulphur-dioxide gas in solution tends to form sulphuric acid, and in practice there is always formed, together with the bisulphite, more or less sulphate which is insoluble and remains in the acid as a white precipitate, commonly called *gypsum*. This precipitate gives a great deal of trouble in the acid and in the digesters. It is generally regarded as insoluble, but this opinion is not absolutely justified, and a sufficient amount of it dissolved in the acid afterwards separates out in the digester, where it frequently plugs up in the bottom connections of the digester, causing trouble when the digester is to be blown.

52. The removal of the gypsum is a matter of great difficulty, for the reason that when it is allowed to accumulate it has to be chipped out with a hammer and chisel. This is a troublesome and expensive process and involves shutting down the digester; it is also hard on the lining. Sometimes the gypsum will close up the outlet from the digester to such an extent as to make it very slow and difficult to blow out the stock. From the considerations stated it will be realized that every precaution should be taken to prevent the formation of sulphate, or gypsum, in the acid. This can be avoided only by accurate chemical control of the acid plant and by constant watchfulness on the part of the entire operating force. The cause of sulphate, or gypsum, in the acid may, as can be seen from the preceding consideration, lie with the operation of the sulphur burners, or the coolers, or the milk-of-lime or tower system. If difficulty is found in keeping the acid free from sulphate, all of the preceding possibilities should be carefully investigated.

53. Tower Systems.—The most efficient tower system is the Jenssen, the towers of which are constructed of reinforced concrete, lined with acid-resisting tile. The following is a

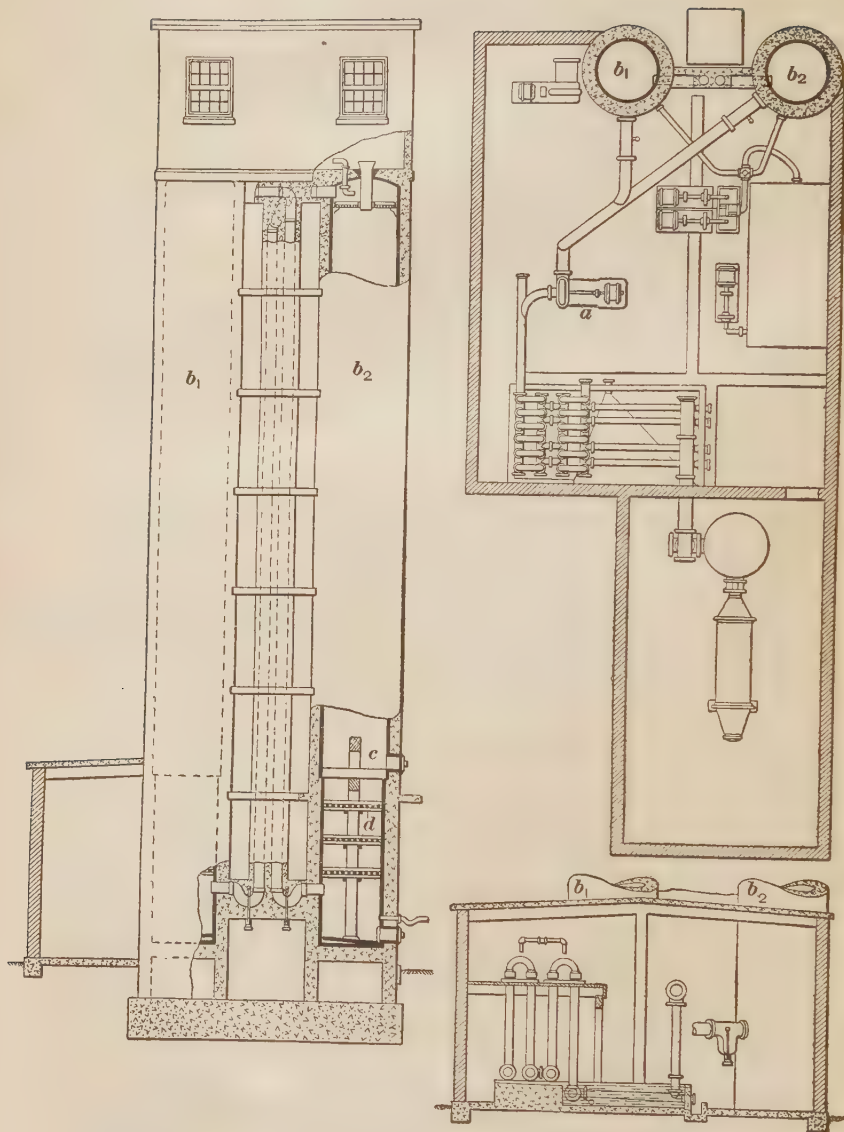


FIG. 9

description of a typical installation which is illustrated in Fig. 9. A hard-lead fan *a*, directly connected to a variable-speed motor so as to allow the capacity of the plant to be carried at any desired point, blows the sulphite gas through the concrete towers *b*₁ and *b*₂, which work in series. After the gases have passed through tower *b*₁ they enter tower *b*₂ at the bottom through a tile pipe. The unabsorbed gases (carbonic acid, nitrogen, and oxygen) pass out into the air through a pipe on top of the second tower in which is formed a very weak acid, and which is pumped into the first tower, where the finished acid is made. The grates *c*, on which the limestone rests are elevated from 17 to 20 feet above the gas inlets, and the space between the gas inlets and these grates is filled with a wooden checkerwork fixture *d*. The reason for this arrangement is to allow the acid to be saturated with free SO_2 before the gas strikes the limestone. Careful experiment has shown that from 17 to 20 feet of wooden checkerwork is sufficient to bring the acid to the point of saturation. Raising the grate still higher would not influence the content of free SO_2 to any extent. By arranging the tower in this way, it has been possible to produce with 15 or 16 per cent. gas and a water temperature of 5° C. a tower acid containing 4.5 per cent. total SO_2 , and 3.1 to 3.2 per cent. free SO_2 . Such an acid in connection with a proper reclaiming system and proper operation of the digester relief brings the percentage of total SO_2 in the cooking acid up to 7 per cent. or higher.

54. When the towers have been in operation 2 or 3 days they are reversed, the second tower being used as the first, and vice versa. This allows the first tower to be charged with limestone as the cover on top can be removed after creating a slight vacuum with a steam jet, the gas in this way being prevented from entering the charging room. The high efficiency of this system is demonstrated by the fact that over 90 per cent. of the gas is absorbed in the first tower, the towers being operated under forced draft and with a large volume of water. For this reason, it has been found that cleaning the limestone grates is a thing of the past. The system can be shut down on

Sunday morning and started up on Monday without any cleaning whatever. The foregoing arrangement of the towers is used where a straight calcium limestone not exceeding 8 to 10 per cent. magnesium carbonate is at hand.

55. Where a limestone or dolomite containing as high as 40 to 50 per cent. magnesium carbonate is to be used, the tower construction has to be changed. It is well known that calcium carbonate is easier to dissolve than magnesium carbonate, and, due to this lack of uniformity in the solution of magnesium and calcium carbonate, the latter is first attacked by the gases, with the result that the surface of the stone crumbles and a sludge is formed. In order to dissolve this sludge, the grates, when a limestone high in magnesia is being used, should be elevated 40 feet above the gas inlets, and four towers should be used in order to obtain sufficient capacity to cover the slower absorption ability of the high magnesia stone.

56. Reclaiming Systems.—Recovery towers are the chief feature of the reclaiming system. These are usually of concrete and may be filled with wooden checkerwork or with logs or with chemical stoneware rings. Dilute acid from the tower system is pumped into the top of the reclaiming tower and flows down the tower, meeting and absorbing the gas from the relief lines of the digesters. The liquid never absorbs all the gas and the unabsorbed gas fills the space in the tower above the liquid. This makes a pressure of gas on the liquid which enables it to hold more gas in solution than if air were present above the surface of the liquid. The reclaiming of the relieved liquor is very simple, as it is simply run to tanks, from which it is mixed with the raw acid. The gas, however, has to be cooled before going to the reclaiming towers. The coolers are similar to those used for the cooling of the gas from the burners. The effect of temperature on the strength of the acid is very marked. Whereas, at 0° C., 1 volume of water will dissolve approximately 80 volumes of sulphur gas, at 40° C. it will dissolve only about 19 volumes. Consequently,

cold water should be used in the towers, the reclaiming system should be kept cold, and all pipe lines containing raw or cooking acid should be laid out with this end in view. In some mills refrigerating systems have had to be installed to keep up the strength of the free acid in summer.

57. Preparation of Wood.—In the sulphite process, knots, pieces of bark, and decayed wood are scarcely acted on by the acid liquor, and should therefore be removed before the chips are sent to the digester.

The work of preparing the wood is carried out in a part of the mill called the *wood room*. The wood room is a system of machines, conveyers, etc., for cleaning wood, for removing bark, and in those mills where the pulp is to be manufactured by a chemical process (sulphite, sulphate, or soda process) for chopping and screening the wood into designated sizes for the digesters. In the case of mills making ground wood or mechanical pulp, the blocks are delivered direct to the grinders from the wood room. In some mills the bark is removed in tumbling drums and in others by means of *barkers* or *barking machines*. The latter are less efficient both from the standpoint of labor and waste. Tumbling drums will save sometimes 50 per cent. of the labor and 10 per cent. of good wood which the barking machine wastes. However, the first cost of the equipment is much higher. By means of a chain or cable conveyer, 2-foot or 4-foot blocks are delivered to the wood room.

58. On arriving at the wood room, the wood is usually dumped from the conveyer into a pond or a tank of water. A centrifugal pump usually takes water out of the lower end of the tank and delivers it to the head end, thus causing a continuous current, which floats the logs toward the tumbling drums or barking machines. There are several reasons why this floating process is preferable to mechanical conveyers. First, the maintenance of a floating process is very small compared with other methods; second, the wood in winter time is freed from ice, and the dirt attached to the ice, by heating

the water with exhaust steam; third, all dirt adhering to the wood is pretty sure to be removed during its progress through the pond; fourth, it serves as a storage reservoir holding a surplus of wood so that there will always be the needed supply on hand for the chippers, or grinders. This pond is usually from 3 to 4 feet deep, and is made of concrete.

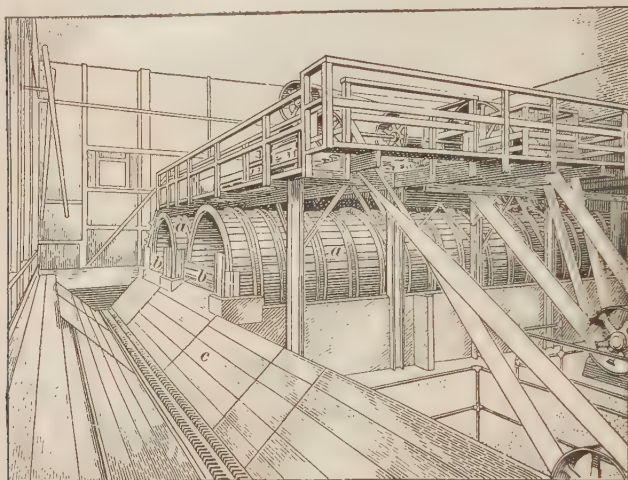


FIG. 10

59. Tumbling Drums.—Tumbling drums *a*, Fig. 10, are made of steel channel bars open at each end and operating continuously, the logs being fed into one end of the slightly inclined drums, and, after having all the bark rubbed off by jostling against one another and the drum, gradually working out of the other end. The drums dip in a pond of water which is scooped up by the channel bars. Water pours over the logs constantly while in the drums, and washes away the bark, which accumulates in a waste pile and is generally burned. From the lower end *b* of the tumbling drums the blocks fall on a conveyer *c* that takes them to the chippers, in the case of sulphite mills, etc., or to the grinders in the case of ground-wood mills. A man is stationed at a convenient point who transfers to a conveyer, leading from the pond in the wood room through the drum for rebarking, all logs from which

the bark has not been completely removed. The usual size for the drums is 9 to 10 feet diameter by 25 to 30 feet long, and the capacity is 12 to 15 cords per hour of 4-foot wood. About 100 horsepower is required to drive a drum 10 feet by 30 feet.

60. Barking Machines.—Although barking machines are more wasteful of wood than barking drums, and although it requires more labor to operate them, they are still ordinarily found in American mills. The first drum installations in this country were not conspicuously successful, and the fact that improved equipment has removed the causes of the early failures has not yet become as universally appreciated as might be desired.

A barking machine works on the same principle as a carpenter's plane. Four knives analogous to the blades of the plane are set, equal distances apart, in a heavy, round, flat-faced revolving disk. If a block of wood is held against these revolving plane knives, the wood or bark will be shaved from the block. Turning the block will thus cause the bark to be removed from the entire circumference.

61. Chippers.—Blocks 2 feet or 4 feet long, free from bark are brought by a mechanical conveyer from the tumbling drums or barking machines to the chippers. These are powerful machines, one of which is illustrated in Figs. 11 and 12, and will slice the blocks crosswise of the grain into sections $\frac{1}{8}$ of an inch thick, at the rate of 800 slices per minute. Before explaining the construction and operation of the machine, it may be well to explain why the blocks are not sawed into sections of the right thickness. The method has been tried, but has been found too wasteful and too slow.

The modern *four-knife chipper*, shown in Fig. 12, consists of a flat-faced disk *a* of solid cast iron on the rim of which a steel ring is shrunk for safety. The disk is about 84 inches in diameter, 4 inches thick, and weighs about 3 tons. It is run at a speed of from 200 to 225 revolutions per minute. The disk is firmly keyed to a shaft *b* that passes through its

center. On either end of this shaft are journals supported in journal bearings *c*. Keyed to this same shaft is a driving pulley *d* of suitable dimensions, by which the disk is driven. The disk shaft and pulley thus assembled are all mounted in a very substantial cast-iron frame *e*, designed to stand the violent hammering and strain. The frame is provided with a spout *g*. The disk, like the barking machine, has three or four knives *f* placed in a circle. The knives are much stronger than the knives on the barking machine and are ground at

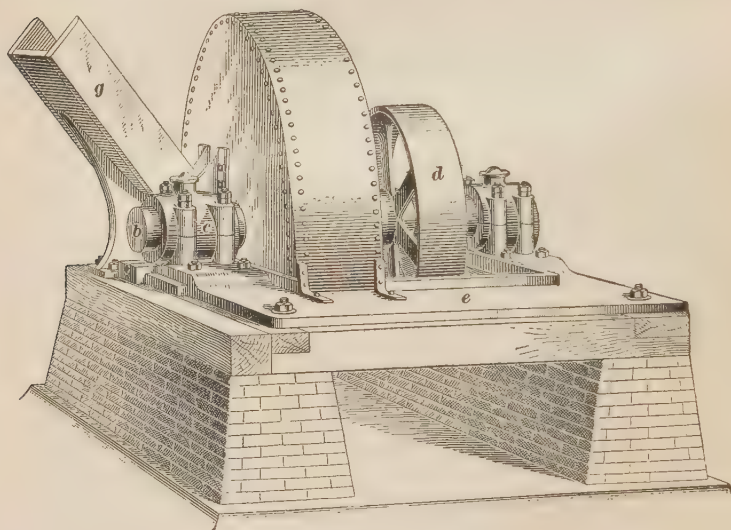


FIG. 11

an angle varying with the make of the knife and regulated by the quality (whether wet or dry, etc.) of the wood to be chipped. The product of the revolutions per minute and the number of blades gives the number of cuts per minute, and knowing that the chips are always $\frac{7}{8}$ inch thick, and knowing the number of sticks to a cord, it is possible to calculate the theoretical capacity of a chipper. Allowance should, of course, be made for the efficiency of the feeding system.

62. Crushers.—The crusher is a machine for disintegrating the chips into specified weights and thicknesses. The usual

type consists of a rotor to which are attached swinging pins, which are held reasonably positive by the action of centrifugal force. The pins are held on pivots of lignum vitae or oak, thereby preventing undue binding and permit the pins to be thrown back in case a spike or a bolt inadvertently passes through the crusher, thus saving the machine from being wrecked. The pins should be maintained complete and in good condition. Operating the crusher with missing pins permits coarse chips and knots to be delivered to the belt,

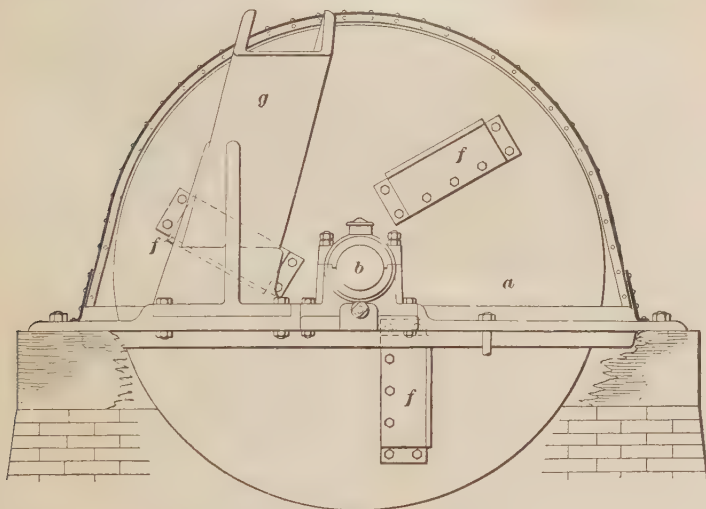


FIG. 12

and, while the screens will reject these to the refiner, the efficiency of the whole installation will be lowered on account of the excessive amount of waste of poorly prepared chips, etc., rejected by the screens. The usual speed of the crusher is from 2,000 to 3,000 revolutions per minute.

63. Screens.—The chips must be screened very carefully, not only to eliminate large chips that have failed to be disintegrated in the crusher, knots, etc., but also to separate too finely divided particles, sawdust, etc. The chips from the crushers are elevated to the screens by means of a scraper or

drag conveyer that passes beneath the crusher and receives the chips from the crusher through a hopper. Several types of screens are in use. The *Lombard*, or *rotary type* resembles a tumbling drum. The first section consists of a fine sawdust section, removing all sawdust and particles smaller than the standard chips. The second section consists of a coarse mesh screen with perforations approximately 1 inch in diameter, permitting the standard chips to fall through and ultimately go to the digester, and rejecting the balance. The chief objection to this type of screen is that long slim chips, which should go to the rechipper, stand on end and go through. The

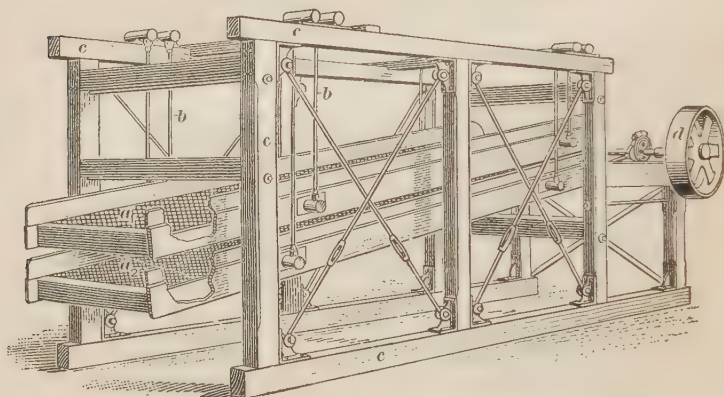


FIG. 13

shaker-type screen is preferable and works more efficiently. The shaking motion has a tendency to hold the slivers parallel to the screen and sift them off.

64. The shaker screen, illustrated in Fig. 13, consists of two large, flat trays *a* and *a*₂, one directly above the other suspended by rods *b* from a wooden frame *c* and vibrated by a pulley *d* actuating an eccentric. The top tray is covered with perforated metal or wire, with openings about 1 inch by 1¾ inches in size, which let out the chips, but retain the large knots and slivers. The motion of the screen moves these rejected particles to the discharge end, where they fall into a tank of water. The slivers and large pieces of sound wood

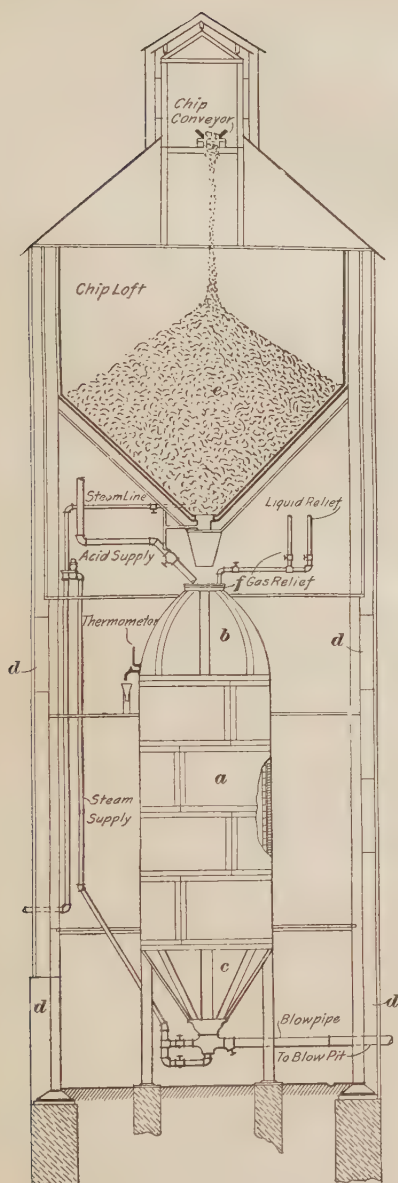


FIG. 14

float and are skimmed from the top of the water by a conveyer chain, passing into the rechipper. The chips from the rechipper then pass back to the screens. The knots, being heavier than water, sink in the tank and are removed from the bottom at intervals. Knots are useless as material for pulp.

The bottom tray of the screen is covered with perforated metal having openings only large enough for sawdust and fine particles of dirt to fall through. This waste is automatically conveyed to the boiler house for fuel.

The standard sized chips, freed from those particles that are either too large or too small, fall from the screen to a belt conveyor by which they are carried to the chip bins.

65. Digesters.—The typical form of digester used in the sulphite process today is illustrated in Fig. 14. It consists of a tall cylindrical vessel *a* of steel-plate construction with a dome-shaped top *b* and a conical bottom *c*. The

size of the digester depends on the method of cooking the wood. The steel plate is usually $\frac{7}{8}$ inch to $1\frac{1}{4}$ inches in thickness, and the joints are triple riveted.

The digesters are located in a tall structure *d* that supports the chip bin *e*.

The digesters rise through two floors, one floor being placed just where the cylindrical portion begins and the other being located so that the digester heads *f* just rise through it. The digesters should stand alone, no floors coming in contact with them. The digester house should be strongly constructed, well illuminated, and accessible at all levels by good stairways. It should also be provided with a good system of ventilation to carry off the fumes.

A digester of the shape described, 15 feet in diameter and 49 feet high, will hold approximately 27 cords of chipped wood and 28,850 gallons of acid. The essential parts of the digester are: the shell; the lining; an opening, provided with a cover at the top for receiving chips; a system for filling the digester with the acid; a system of pipes for supplying the steam for cooking; a system of relief line for getting rid of steam and acid gas; thermometers to record the temperatures; gauges for recording the steam pressure; and a blow valve and blow pipe through which the pulp is discharged under pressure when the cook is completed.

66. Lining.—After experimenting with a great variety of linings, such as lead, tiles, etc., pulp-mill engineers have almost universally adopted the brick lining for sulphite digesters. There are generally two courses or layers of brick, the first or outer course being separated from the steel shell by a 1- to 2-inch layer of cement composed of Portland cement, crushed quartz and fireclay. The bricks are laid with a cement made of one part of sand or crushed quartz, and one part of litharge, mixed dry, and glycerine added to form a thick paste. The glycerine should be slightly warmed before it is used. Sometimes a little silicate of soda, which helps the cement to set well, is added to the glycerine. After the glycerine is added, the cement is kneaded like bread till it is of an even consistency. The

glycerine should be added to small portions of the dry ingredients at a time, the cement thus being prepared about as fast as it can be used. A ball that a man can nicely hold in both hands will lay about five bricks. Frequently the first layer of brick is common red brick and the inner layer special vitrified acid-resisting brick. The two layers are separated by a thin layer of cement mortar. The inner layer of vitrified brick is laid with great care in the litharge cement just described.

When a digester has been shut down to be relined, or for any other cause, or is being started for the first time, so that it is cold, it should not be heated to a high temperature suddenly. The brick should be allowed to dry out thoroughly and then the digester gradually brought up to a cooking temperature. The digester should be at least 24 to 36 hours in warming up from cold to ordinary cooking temperature.

67. Digester Head.—The head or cover of a digester is removable. It is fastened to a casting that forms the top rim of the digester proper, by means of clamps which hold it securely in position against the internal pressure of the digester. The head is lined with a sheet of some acid-resisting metal such as aluminum. Between the head and the rim on which it sits, is inserted a gasket, which has usually been found economical and efficient to make out of a lap of sulphite pulp. A chain hoist is provided for removing and replacing the digester heads, which have to be removed every time the digester is filled with chips and acid.

The head is fitted for the connection to the relief line, which is usually a 2½-inch bronze pipe. It relieves the pressure in the digester after the steam has been turned on and allows the gas that forms in the top or neck of the digester, to escape to the recovery system. After leaving the digester, this line divides into two separate lines, one a liquor-relief line and one a gas-relief line. These lines are controlled by valves and the attendant can tell by the sound whether liquor or gas is coming off from the digester and thus direct it into the proper line. Both of the lines are of bronze and usually 2½ inches in diameter. Strainers should be provided to prevent

pulp from getting into the relief lines, thus plugging them up. They are usually hard lead or bronze globes or hemispheres provided with numerous holes about $\frac{3}{16}$ inch in diameter.

68. Steam Supply.—At the base of the digester are the inlets for the steam. The connections between the boilers and the digesters should be as short as possible to prevent condensation of the steam and reduction of the steam pressure. Suitable traps should be provided and all lines should be covered with an efficient heat-insulating substance such as magnesia.

Where a number of digesters are used, as is usually the case, the piping system should be designed so that all the digesters will receive steam at the same pressure, which will not be the case unless the main line leading from the boilers is adequate in size.

The steam pipe for the digester is usually carried up to a point near the top of the digester and then down again, entering the digester at the bottom. This forms a trap which prevents anything from being forced from the digesters back into the boiler system, should the steam pressure for any reason become less than the pressure in the digesters. This arrangement permits the reducing valve to be located on the working floor, that is, the floor where the digester heads, gauges, etc., are located, and thus be under the eye of the operator at all times. This line should be provided with one or more suitable check-valves, one in the horizontal section of the pipe accessible to the working floor. The location of the second, if such is installed, is between the first check-valve and the digester.

To the bottom of the digester is fastened a bronze **T**, the side outlet being attached to the digester. To one end of the **T** the 4-inch bronze steam line is connected. It is of bronze to resist any action on the part of any acid from the digester getting into it. To the other end of the **T** is fastened a bronze blow-off valve, and to this a 10-inch cast-iron pipe increasing in diameter to 12 inches, at the farther end, and passing through the side of the blow pit.

69. Cooking.—There are in general two methods of cooking sulphite pulp. That in general use is the *direct cook*, sometimes known as the *Ritter-Kellner process*, which is carried out in the vertical digester already described. The other and older method, which is still used for certain grades of pulp is the *Mitscherlich process*, which is an indirect process, the steam never coming in direct contact with the pulp. The Mitscherlich process requires a special form of digester fitted with steam coils.

70. Direct-Cooking Process.—The first advantage of the Ritter-Kellner process is the fact that it is quick, the time varying in different mills, but never being nearly as long as for the Mitscherlich process. The second great advantage is the comparative simplicity of the equipment, leaks and other causes of stoppage being cut down to a minimum and repairs being as low as possible.

In charging, the digester is filled as completely as possible with chips which, before the cooking operation has proceeded very far, will have settled enough to be covered with the liquor.

A digester 15 feet in diameter by 49 feet high will hold approximately 27 cords of chipped wood and require about 28,850 gallons of liquor. Such a digester will produce about 12 or 13 tons of air-dry pulp.

The liquor should be run in as quickly as possible. The pipe for this purpose should be at least 6-inch and may be larger. Like all other connections with which the acid comes in contact, it is of hard lead or bronze. An 8-inch centrifugal pump of hard bronze is satisfactory for pumping the acid into such a digester. Running at approximately 850 revolutions per minute, it will fill the digester in 25 minutes. It requires 24 horsepower against a 60-foot head. All connections from the tank to this pump and from the pump to the digester can best be made of hard lead containing in its composition 8 per cent. of antimony. Modern mills are frequently designed so that the acid flows into the digester from tanks placed at a higher level than the digesters, thus obviating the use of such a pump.

71. In some mills the acid is pumped into the bottom of the digester, a check-valve being inserted to prevent the acid from running back in case of accident to the pump, this valve being closed before stopping the pump. Thus the acid rises gradually through the chips, and fumes are avoided. However, the method is not to be recommended, as the chips float and do not become thoroughly saturated with acid, and it is hard to tell when the digester is full. When the digester is filled with chips and acid, the cover is securely bolted on and the pressure- and temperature-recording instruments are noted, and at times calibrated. The relief valve is adjusted slightly to let out the air. The steam valve is then opened and the digester allowed to come gradually to 65 or 70 pounds pressure. It is very important that the pressure shall not be applied too suddenly. The acid should be allowed to saturate the chips thoroughly and penetrate every particle before any notable increase in temperature takes place. This is necessary to produce the proper chemical changes in the material and to prevent charring and overcooking of the chips.

72. If the heating is forced at the beginning of the cook, the steam striking the cold liquor will hammer, which is hard on the digester. However, the chief reason for raising the temperature slowly is the effect on the chips as just mentioned. If reddish or brown bundles of fiber are found in the pulp, it is an indication that the pressure has been applied before the acid had a chance to saturate the chips thoroughly.

The pressure carried throughout the main portion of the cook depends on the grade of stock required, physical conditions of the digester, maximum pressure allowed by the insurance companies, etc. In general, the temperature should not be allowed to exceed 110° C. until 2½ hours have elapsed. When the steam pressure has been brought up to standard, the relief valves are opened sufficiently to shake up the contents and bring the acid up over the top of the chips.

73. It is impossible to give any general directions for cooking. The cook varies in every mill, depending on the materials being used, the grade of pulp to be produced, and the

personal ideas of the man in charge of operations. In some mills very elaborate records are kept of the temperature and pressure throughout the cooking operation, as well as of the chemical composition of the samples drawn off at various stages, and careful deductions are made from these facts and the digesters operated accordingly. In other mills, unfortunately, rule-of-thumb methods prevail and the operation of the digesters depends entirely on the idiosyncrasies of some one man. If the grade of pulp to be produced has been settled, the question may be asked why the strength of the acid, the temperature, the pressure at every stage of the cook, and the duration of the cook cannot be absolutely standardized and the operation made to conform to these standards. The chief factor preventing this is the wood, which will always vary in moisture content and in other respects. Such variable factors make it necessary for the cooking operation to be in charge of a capable and experienced man who can exercise judgment.

74. Mitscherlich Process.—The pulp produced by the Mitscherlich process is of excellent quality, but the process has the following disadvantages: Slowness, the cooking time being very long, usually 35 to 40 hours and in some cases upwards of 60 hours, the pressure rarely exceeding 45 pounds; the digester has from 1,000 to 3,000 feet of lead or copper pipe coil in it for heating purposes which is constantly in need of repair, and when the heating coils have to be repaired the digester has to be shut down; on account of leaks, the acid liquor gets into the condensate from the steam used for heating, thus making it impossible to use this condensate in the boiler plant and thus increasing the consumption of coal; calcium monosulphite collects on the lead pipes and has to be removed and will frequently drop into the pulp during the cooking, where it is very undesirable; the chips are brought into direct contact with the heating surface, frequently causing overcooked pulp and lower yield from the wood; the pulp cannot be blown out of the digester, but it is removed by shovels and rakes through a large manhole near the bottom of the digester after the pressure has been relieved.

In spite of all drawbacks, the Mitscherlich process is found

advisable in some cases. It makes a very strong pulp, ideal for certain purposes, which cannot be exactly imitated with a direct cook. It is also economical of sulphur. A much weaker acid can be used than is customary with the direct cook.

75. Washing the Pulp.—In the Ritter-Kellner process, when the cook is finished, the stock is blown from the digester through the blow pipe into the blow pit, a vertical tank of wooden or concrete construction of sufficient capacity to hold two blows, that is, twice the capacity of the digester. It is strongly reinforced with iron hoops, if of wood, and has a cast-iron plate or target placed on the side opposite the blow pipe. This is to protect the tank from the violent pounding of the stock as it is blown from the digester under pressure. The blow pit has a chimney-like opening at the top containing baffles to prevent any stock from being forced out. This is called a *vomit pipe*.

The bottom of the blow pit is equipped with a false or second bottom, which is usually of 3-inch matched plank, set approximately 6 inches above the actual tank bottom, and provided with holes approximately $1\frac{1}{2}$ inches from center to center. These holes are usually $\frac{1}{8}$ inch in diameter at the top and flare to $\frac{1}{2}$ inch in diameter at the bottom. Sometimes the false bottom is made of perforated vitrified-brick tile set on planks provided with large holes and held in position by wooden fillers wedged between the tiles.

76. The purpose of the false bottom is to permit of washing the stock after it has been blown into the pit. The pit is filled with water to a height of 2 or 3 feet on the stock. The water filters down through the stock and passes through the perforated bottom, carrying much of the acid with it, to the sewer or a save-all. This operation is repeated until the stock is washed free from all acid.

The blow pit is fitted with a lead-pipe connection at a point just above the drainer or false bottom which leads to the centrifugal pump used to pump the stock from the pit to the intermediate stock chest.

The stock, as it is left in the blow pit after washing, is of a consistency of about 12 to 15 per cent. air dry, and in order to get it out it is necessary to use water to thin it down. This is done with a high-pressure hose line, the operation being called *hosing*, or *sluicing*.

77. Intermediate Tanks.—The object of the intermediate tank is to supply a storage between the blow pit and the screens. This tank has a capacity of approximately 2 or 3 tons, and is equipped with an agitator. Such tanks make certain that the stock will be of a much more uniform consistency than if it were pumped direct from the blow pit to the washers and screens. From the intermediate tanks the pulp goes to the worm-washers and screens.

78. Worm-Washer.—A very convenient form of washer, known as the *worm-washer*, is shown in Fig. 15, in which (*a*) is an elevation of the end at which the washed stock is delivered, and (*b*) a longitudinal section on the line *AB* in (*a*). The washer consists of two copper worms *a*, enclosed in perforated copper cylinders *b*. The cylinders are partly enclosed by a box *c* that catches the wash water and conveys it away through the pipes *d*. There is a support *e* at one end of the box, through which the hollow trunnions *f* of the cylinder pass. There is a large sprocket wheel *g* at the end of each trunnion, and an endless chain connects the teeth of the two wheels, so that they are made to revolve at the same time. The stock enters the cylinders through the pipes *h* that pass through the hollow trunnions, which revolve around the pipe. There are heavy iron bands *i* encircling each cylinder at intervals, and at the discharge end each cylinder is encircled by a heavier band, or ring *j*, which passes through the hollow wheels *k*, and thus holds the cylinders steady while revolving.

79. A perforated water pipe *l*, Fig. 15, extends along the whole length of each cylinder, and forcibly emits small jets of water against them. The cylinders are perforated, and the jets from the pipes *l* are emitted only on the surface, which is directly over the box. This water penetrates the cylinders

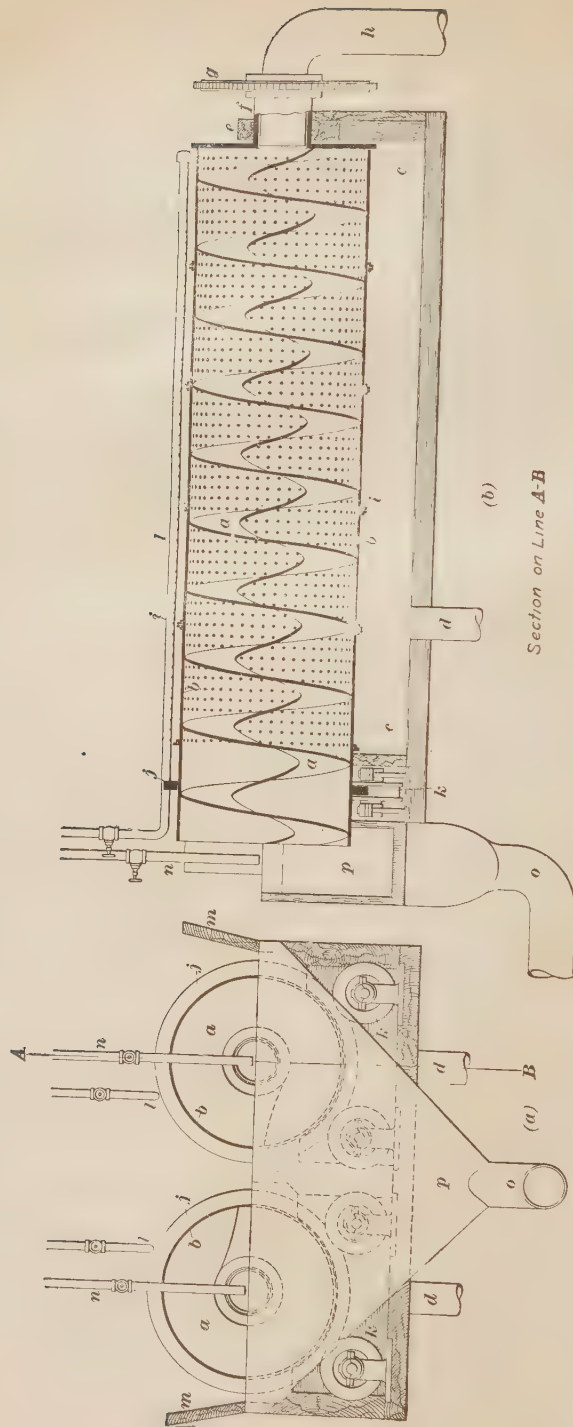


Fig. 15

and washes the pulp during its passage through the worms. Dashboards *m* extend from the sides of the box and are about as high as the cylinder. These dashboards prevent the water from splashing out.

At the discharge end of the cylinders there is a funnel-shaped box *p*, into which the washed stock from the two cylinders is deposited and washed down by water from the pipes *n* into the exit pipe *o*. The waste-water box *c* is hollowed out at this end, so as to allow the drums to pass through. In its passage through the worms the stock is turned over and over, thus exposing all surfaces to the washing action.

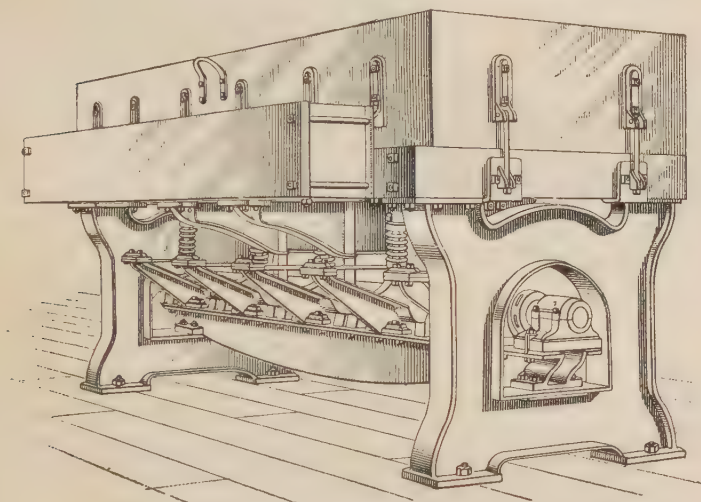


FIG. 16

80. Riffler.—The stock in passing from the worm-washer to the next set of screens (which are either centrifugal or diaphragm screens) passes through what is called a *riffler*. This is a shallow wooden sluice about 6 or 8 feet wide, equipped with a set of baffles all along the bottom. The riffler is on a level without any incline, so that the current is leisurely being produced by the pump that draws the stock from the riffle. In this device the various impurities that may be in the stock sink to the bottom and are held by the baffles. The riffler should

be about 18 inches deep and the baffles about 8 inches high and 8 or 9 inches from each other. The capacity of the riffler should be sufficient to take care of the output of the digesters and the length is governed chiefly by the amount of space able to be devoted to it, the longer the better.

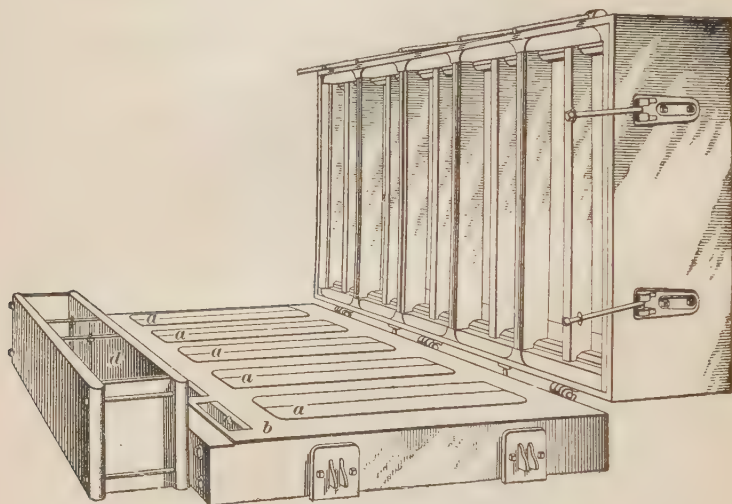


FIG. 17

81. Diaphragm Screens.—Diaphragm screens are of various makes but all have about the same form and accomplish

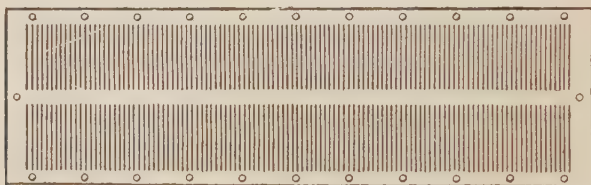


FIG. 18

the same purpose by making use of the combined action of gravity and suction on the stock. Figs. 16 to 19 illustrate the usual diaphragm screen and its parts. Fig. 16 shows a general exterior view of the screen box and the flow box, together with the vibrating mechanisms. Fig. 17 shows the screen

box open, exposing the wooden diaphragm plates *a*, the rubber diaphragm *b*, and the apertures *c* through which the stock passes in going to the flow box *d*. Fig. 18 shows one of the screen plates. The slits in this plate are made very fine so as to permit the passage of only well-cooked fiber, to hold back material that has not been acted on, etc. These screen plates should be watched very carefully, and when there are any openings large enough to allow this slivry substance to pass through, the plate should be changed. Screen plates that have

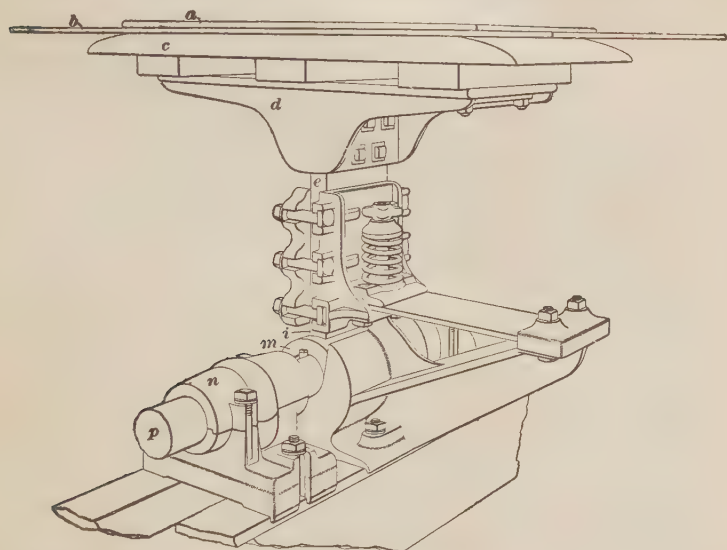


FIG. 19

thus become worn and removed are generally used as *second screen*. The slots in these plates can be closed up and recut one or twice.

When operating the screen, a section of which is shown without the screen box in Fig. 19, the stock is pumped into one end of the screen box, Fig. 17, in the form of a thin watery suspension containing about one-half of 1 per cent. of pulp, and flows toward the opposite end over the screen plates contained in the upper part of the screen box. As the stock flows over the screen plates, that part of it which is desirable passes

through the slits in the screens aided by the suction created by the action of the rubber diaphragm. The manner in which the diaphragm is made to function is made clear by a study of Fig. 19. The diaphragm *b*, which is held in place by the wooden clamps *a* and *c*, is given a vertical vibratory motion by the table *d* and the plunger *e* which are actuated by a cam *m*. This cam is square-shaped with rounded corners and revolves with the shaft *p* supported by the journal *n*.

82. For every revolution of the shaft there are four upward and four downward movements of the shoe *i*. These movements are communicated to the rubber diaphragms, which give the screen its suction. When the shaft is running at full speed, it makes from 165 to 175 revolutions per minute, which causes from 660 to 700 vibrations of the diaphragm per minute. There is a space of 4 inches between the screen plate and the top diaphragms, so that at each downward stroke the pulp is sucked through the screen plate into this space; from here it runs through the apertures *c*, Fig. 17, at the side of the diaphragms and out through the flow box. The flow box is provided with a partition that causes the outflowing stock to stand at such a level that the apertures in the diaphragms are always covered; otherwise the screen would lose its suction, by air coming back through the flow box, and refuse to act. In case the screen refuses to act and flows over, the stock is shut off and, after raising the partition, water is turned into the flow box until it rises to the screen plates in the screen. This operation will drive out all the air, and the screening can then be continued as before.

83. Centrifugal Screens.—The centrifugal screen, illustrated in Fig. 20, consists of a vertical runner or impeller *a* surrounded by a cylindrical screen plate *b*, this in turn being surrounded by a steel-plate shell. The runner is rotated by a pulley *c* and the stock enters through an intake *d*. The stock is urged against the screen plate through ports by the centrifugal force of the runner and the good stock passes through. The rejected stock passes out at the base of the screen *e* and goes to a secondary screen which is of the same construc-

tion, only coarser. The good stock from the secondary screen is usually put back through the system and goes again through the primary screen. Another type of screen operates on the same principle, but it is of a horizontal design.

The entire battery of screens is supplied by a canal-like head-box, with gates opposite each screen intake so that the flow to the screens can be regulated and any particular screen can be cut out at any time. In order to have the stock supply to the screens at a constant head, there is an overflow from the head of the canal-like head-box controlled by an adjustable dam. The overflow leads back to the riffler pump by a return pipe. The stock in the screen head-box is diluted with pure water to a consistency of about .5 per cent. air-dry. The usual type of centrifugal screen runs at about 400 revolutions per minute and has a power consumption of approximately 35 horsepower.

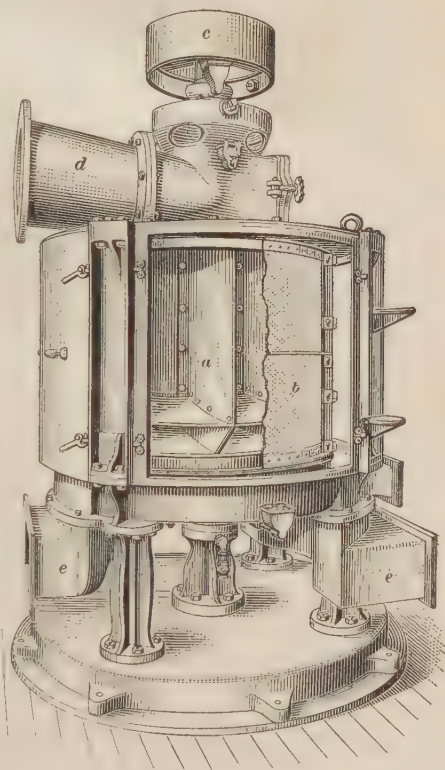


FIG. 20

84. In general, centrifugal screens are used when the quality of the paper to be produced will tolerate their comparative incomplete screening action. In the manufacture of fine papers, diaphragm screens are used.

Centrifugal screens are kept clean by means of a specially designed portable shower on the end of a hose which can be inserted between the screen plates and the outer shell. The pressure on this shower should be not less than 30 pounds, as keeping the screen plates clean is a very important factor in operating centrifugal screens. The size of the screen plate openings may vary from .055 to .1 inch, depending on the grade of stock being screened.

The good stock from the screens goes either to deckers or wet machines, as already described in connection with the mechanical process of the production of pulp. The forming of the pulp into laps is the same for all processes by which pulp is prepared.

SODA PROCESS

85. Nature of Soda Process.—The soda process is much older than the sulphite process, and is of itself much simpler; however, in order to carry it on at a profit, it is necessary to recover the soda used in the process. This is a somewhat complex operation, and most of the problems of soda-pulp manufacture are connected with the operation of the recovery department. The process depends on the fact that a caustic-soda solution at high temperatures will dissolve all the other constituents of wood except the cellulose, leaving the latter in a form suitable for paper manufacture.

Soda pulp is necessary for certain kinds of paper. Also, the soda process will serve to utilize many kinds of wood that cannot profitably be dealt with by the sulphite process. Poplar is the principal kind of wood used for soda pulp; but some spruce, pine, hemlock, cottonwood, etc., are also worked up by this process.

86. Preparation of Wood.—In general, the wood is prepared for the soda process exactly as described for the sulphite process. However, it is not necessary to exercise quite the same care to get rid of all the small particles of bark, knots, and decayed wood; the soda liquor readily dissolves such impurities. Also, the chips are usually made a little

smaller for the soda process, being from $\frac{3}{8}$ inch to $\frac{1}{2}$ inch in length.



FIG. 21

87. Digesters.—In the older mills, spherical or horizontal rotary digesters, similar to those used for boiling rags, were used. These were heated with steam coils, the steam passing

through the trunnions, and the coils rotating with the digester. These digesters were of small capacity and the cost of up-keep was high.

Such rotary digesters were soon replaced by stationary vertical digesters. The earlier digesters of this type, which are still in use in many mills, were usually from 26 to 29 feet high and 7 feet in diameter, holding about $3\frac{1}{2}$ cords of chips and 4,000 gallons of liquor. As shown in Fig. 21, the top *a* is dome-shaped, and contains an opening *b* through which the chips and liquor are charged. The steam line *c* enters the top and extends to the bottom, where it discharges through a steam ejector *d*, so constructed as to keep the liquor in constant circulation. At the bottom there is a perforated plate, or cone, *e* that surrounds the outlet *f*, but does not cover it. The liquor passing through this plate is conveyed by the ejector and the circulating pipe *g* to the top of the digester and again discharged over the chips, this circulation being maintained throughout the cooking operation. The digester is discharged through an outlet pipe *h* controlled by a valve *i*.

The size of the digesters has been increased, and digesters as large as 65 feet high by 19 feet wide have been used. Various systems for circulating the liquor have also been introduced. One of the most important of these is the Morterud process, whereby the liquor is heated in a separate heater and pumped through the digester. This process has the advantage that the liquor is not diluted by the use of direct steam.

88. Digesting or Cooking.—One of the smaller digesters just described holds about $3\frac{1}{2}$ cords of wood. The liquor used is caustic-soda solution having a strength of 11.5° to 12° Baumé at 60° F. and a causticity of from 92 to 95 per cent. For such a digester about 4,000 gallons of liquor are required. The volume varies with the kind and condition of the wood.

When the digester is full, the lid is bolted on and the steam admitted. The pressure is brought up to a maximum as quickly as possible, and so maintained until the end of the cook. In this regard, this is a much simpler process than the sulphite

process. Pressures of more than 90 pounds were formerly unusual, but in modern mills, from 125 to 130 pounds is the usual range of pressure.

89. The length of the cook depends on the dryness of the wood. The dryer the wood, the more rapidly it absorbs the caustic-soda solution. Usually the cook is completed in from 6 to 8 hours. In order to circulate the liquor, the pressure is relieved from time to time. This causes dilution of the liquor from the new steam entering and loss of heat units and is one of the arguments against the direct cook as compared with the indirect methods where forced circulation is used, because in the soda process there is no reason apart from promoting circulation for relieving the pressure until the cook is completed; that is, no gas is generated during the process which has to be relieved, as is the case in cooking sulphite pulp.

When the cook is completed the valve at the bottom of the digester is opened and the contents blown into a receiving tank. This tank is usually elevated above the wash pans, in which the liquor is eliminated from the pulp, as the digester pressure is sufficient to elevate the pulp to the height of the receiving tank. The steam escapes through a vent at the top of the tank, this vent usually being provided with baffles to prevent any pulp or liquor from being blown or splashed out.

90. Washing the Pulp.—The contents of the receiving tank are next run into the first of a series of wash pans which are provided with perforated false bottoms through which the liquor drains from the pulp. Dilute hot liquor from a certain portion of the recovery system is then sprayed on the pulp in the first pan, which it frees from alkali to a certain extent, at the same time becoming fortified itself. Following this operation more and more dilute liquor and finally clean hot water is sprayed on the pulp long enough to remove the last traces of dissolved intracellular matter and alkali. This water goes to the recovery system also as it drains from the pulp.

The contents of the digester when blown are of quite a different appearance from the contents of a sulphite digester, being dark brown and in some cases black, and having a dis-

tinative empyreumatic odor. The pulp, when the liquor is washed from it, is of a gray or brown color, which it does not lose until it is bleached.

91. The whole principle of washing the contents of the soda digester is to get rid of every last trace of alkali and dissolve intracellular matter thoroughly without diluting the wash liquors any more than is absolutely necessary, as all these liquors have to be evaporated later to recover the soda. Very small traces of liquor left in the pulp render bleaching very difficult and impair seriously the value of the product. Excessive use of wash water makes recovery too costly.

For these reasons the pulp, at each stage of the washing, is washed with the liquor from the pan just before it in series, and the small amount of fresh water used for the final washing of a lot of pulp passes in succession through the whole series of washing pans or tanks (usually four or five in number) in each succeeding one of which the percentage of black liquor retained by the pulp is greater. At the end of the operation the liquor is used to wash the pulp fresh from the digesters, as already mentioned, and as a result of this operation is brought up to a concentration approximating, in properly conducted plants, about 55 per cent. of the original strength. The capacity of the wash pans should be such that no liquor will ever have to be drained to waste. Of course, some soda is unavoidably lost, but if less than 95 per cent. of the soda goes to the evaporators from the pans, there is something wrong in the system.

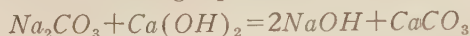
92. The completely washed pulp is removed from the washing pan in exactly the same manner that sulphite pulp is removed from a blow pit. A pump elevates the stuff to the screens. The remainder of the treatment of soda pulp is exactly the same as already described for sulphite pulp. However, diaphragm screens are almost altogether used for soda pulp. Soda pulp is used chiefly for good book and writing papers, hence the use of diaphragm screens in such mills in preference to centrifugal screens.

93. Liquor for Soda Process.—The liquor for digesting the wood in the soda process is made up in pans that are usually about $10\frac{1}{2}$ feet square. The pans are provided with agitators that extend to the bottom and are worked by means of a friction clutch and gearing at the top; also, with siphon pipes that can be raised or lowered and with steam lines running to the bottom. At the bottom of each pan is an opening through which the lime sludge consisting largely of calcium carbonate, is washed into the sewer. When making liquor, this opening is closed with a tight-fitting plug. A water-line, a weak-liquor line, and a line from the leachers empty into each pan, and each of these lines is provided with a cock.

When making a pan of liquor, the leacher-line cock is opened, the recovered sodium-carbonate liquor is allowed to flow in, and the agitator started. The liquor maker from time to time tests the liquor, while it is being strengthened, with a Baumé hydrometer to ascertain its density. If he finds that the liquor is getting too strong, the weak-liquor pump is started and the cock of that line is opened. By practice, the liquor maker is enabled to regulate the supply of the liquors so that there will not be a difference of more than $\frac{1}{4}^{\circ}$ Baumé in the finished pans. If, when a pan is filled up to a certain mark, the liquor does not come up to the desired strength, fresh sodium-carbonate liquor is added to accomplish this end.

94. When using caustic-soda liquor from an electric-bleach plant, as is the case in many of the present mills, this liquor contains a certain amount of salt, which should be allowed for in making the required strength of the finished liquor. For each per cent. of salt there should be an allowance of 1° Baumé, so that, for instance, in making up a $14\frac{1}{2}^{\circ}$ caustic liquor, the strength should be $15\frac{1}{2}^{\circ}$ if the caustic-soda liquor used contains 1 per cent. of salt. When aiming at a certain strength of caustic liquor, the sodium-carbonate liquor should be made about $2\frac{1}{2}^{\circ}$ Baumé stronger than the strength of caustic liquor required. A liquor of good strength is made from sodium-carbonate liquor testing $16\frac{3}{4}^{\circ}$ to 17° Baumé which, when causticized, produces a caustic liquor testing about $14\frac{1}{2}^{\circ}$ Baumé.

If a sodium-carbonate liquor is made stronger than 17° Baumé, it is very hard to causticize. To convert the sodium carbonate to the hydroxide, the liquor is causticized by the addition of lime, which is generally put into an iron cage hanging down into the carbonate liquor. The lime must be added carefully to avoid boiling over. When the required amount of lime has been added, the steam is turned on gradually and the pan allowed to boil for about $\frac{1}{2}$ hour, agitating the liquor continuously. After about a half hour, the agitator is stopped and the sludge is allowed to settle. The conversion of sodium carbonate into caustic soda by the addition of lime takes place according to the following equation:



95. The lime sludge settles to the bottom of the pan, leaving the liquor perfectly clear, provided the *black ash* or recovered ash was well burned. About 8,000 pounds of black ash, containing 48 per cent. Na_2O is used to make a 10½-foot square pan of strong liquor. In practice, from 625 to 650 pounds of good caustic lime is required to causticize 1,000 pounds of soda ash.

It is customary to have two tanks in the cellar under the alkali room, one for storing the strong liquor for the digester room, and the other for storing weak liquor for the second and third washes. This weak liquor is also used in making up the first wash, if necessary, and, when crowded, it is pumped through the leachers in place of water. In some mills each wash is kept in a separate tank and is used in making up the wash preceding it in the next tank.

96. The washes are made as follows: When a pan of strong caustic liquor has settled sufficiently, after the causticizing operation the siphon pipe is lowered and the liquor is siphoned into the strong-liquor tank in the cellar. (It is customary to run a pan of strong liquor and the first wash of another pan at the same time, in order that they will mix in the right proportions in the strong-liquor tank in the cellar.) When the liquor is all out of the strong-liquor pan and the siphon pipe is down to the sludge, the pipe is raised,

the agitator started, and the pan pumped up with a weak liquor by means of a centrifugal pump in the cellar. (It might be well to note here that the centrifugal, or fan pump is used in almost every department.) It is the aim of the liquor maker to make as large a first wash as will be carried by a full, strong-liquor pan, and to produce liquor of the required strength for the digesters. The second and third washes are made similar to the first, and are run down into the weak-liquor tank, to be used as stated. The plug is then withdrawn, and the sludge is washed out into the sewer or over to the lime reclaimer, where it is well drained and burned back to caustic lime so that it can be used again. It is customary in most mills to keep samples of the strong liquor and each of the washes made; these are tested morning and evening by the chemist in charge for Baumé strength, causticity, and per cent. of salt.

97. Recovery Systems.—To clearly understand the recovery process, the composition of the liquor should be remembered. It contains two main ingredients: organic matter in solution, and carbonate of soda. When sufficiently concentrated, the organic matter will burn, yielding a considerable amount of heat which can be utilized in the first stages of concentration of the liquor. At the end of the process there is a mixture of carbonate of soda, unburned carbon, and incombustible mineral matter from the wood (ash), and this mixture goes by the name of *black ash*.

The specific gravity or density of the liquor at the commencement of the recovery process depends on the strength of the original liquor charged, the moisture in the wood, whether the cook was with direct steam or by the indirect method, and the efficiency of the washing system, but is usually from $8\frac{1}{2}$ to 10° Baumé at 60° F. The first attempts at recovery utilized open evaporating pans. Such inefficient equipment was soon abandoned in favor of multiple-effect evaporation in suitably designed equipment.

98. Multiple-Effect Evaporation.—The theory of multiple-effect evaporation is a vast and complex subject, but the

brief explanation of the principles hereafter given may be helpful. The boiling point of water becomes lower as the pressure is decreased, that is, as the vacuum is increased. Thus, it is possible to make water evaporate from a solution being concentrated at a lower temperature, by decreasing the pressure. When water is boiled at atmospheric pressure it boils at 212° F. and the vapor given off has also this temperature. In addition to the heat units it possesses simply by virtue of being at 212° F., it also possesses a large additional number of heat units that have been put into it in converting liquid water into water vapor, or steam. This heat will again be given up when the steam is condensed. It is called the *latent heat of steam*. A pound of steam at 212° F. will have about 1,146 units of heat, of which about 964 will be in the form of latent heat.

99. A multiple-effect evaporator is really a series of boilers, so arranged that the steam from the first boiler, or *effect*, will be carried over and used as the heating agent in the next effect. The second effect is able to boil at a temperature lower than the first, owing to the maintenance of a partial vacuum. The steam from the second effect goes to a third, which operates at a still greater vacuum and thus to a fourth, and so on to perhaps even fifth and sixth effects. Four effects is a usual number in practice. Finally the steam from the last effect goes to a condenser.

Equipment utilizing this principle has been designed with such engineering skill as to make almost perfect utilization of the heat supplied to the machine. The first multiple-effect evaporators used in the paper industry were of the well-known Yaryan type, and although these evaporators have been largely replaced in recent years by equipment of improved design (such as the Scott, Swenson, Zaremba, Buffalo, Badger-Webre, and Kestner evaporators), there are still a good many of them in use and a description will not be out of place.

100. Yaryan Evaporator.—Each effect of the Yaryan evaporator is like a boiler with a number of horizontal pipes arranged in coils parallel to the shell. A supply pipe feeds

the liquor into a header at the back of the machine from which the coils lead. The steam is admitted into the shell surrounding the coils. At the front of the machine the coils end in a header which serves as a separator, separating the vapor from liquor and foam, and in which a partial vacuum is maintained. The difference between the pressure at which the liquor is supplied and the partial vacuum in the front of the machine keeps the liquor constantly moving forwards, forming a film on the surface of the tubes, and in this way a new surface of liquid is constantly exposed to the action of the steam. The liquor from the separator of the first effect is pumped into the second effect and the vapor from the first effect is supplied to the shell of the second effect to give up its heat, and the operation is repeated for as many effects as there may be. The liquor is usually concentrated from its original density to from 32° Baumé to 40° Baumé at 60° F. Of course, the concentration could be carried much higher in modern evaporators, but this is not done because of the difficulty in pumping such very thick liquors. Moreover, the further evaporation of such liquors is not necessary as from 37° Baumé to 40° Baumé has been found a good strength for burning.

101. Disk Evaporators.—Sometimes only part of the water is driven off in multiple effects, the remainder being removed with disk evaporators. This machine employs the principle of exposing a thin film of liquid to direct heat over a large area. This is accomplished by rotating a wheel, or series of wheels, made up of thin steel plates, so that the plates are alternately dipped in the liquid and then exposed to the hot gases which pass through the evaporator in such a manner that they come in contact with the exposed surfaces of all the plates or disks. The moisture is absorbed from the film of liquid and passes in the form of vapor to a stack, or to a reclaiming apparatus, as the case may be. While these evaporators are frequently used alone, they can be used most efficiently to follow a preliminary evaporation in some form of multiple-effect evaporator. In the sulphate process they are usually used alone as the liquors are usually not so dilute

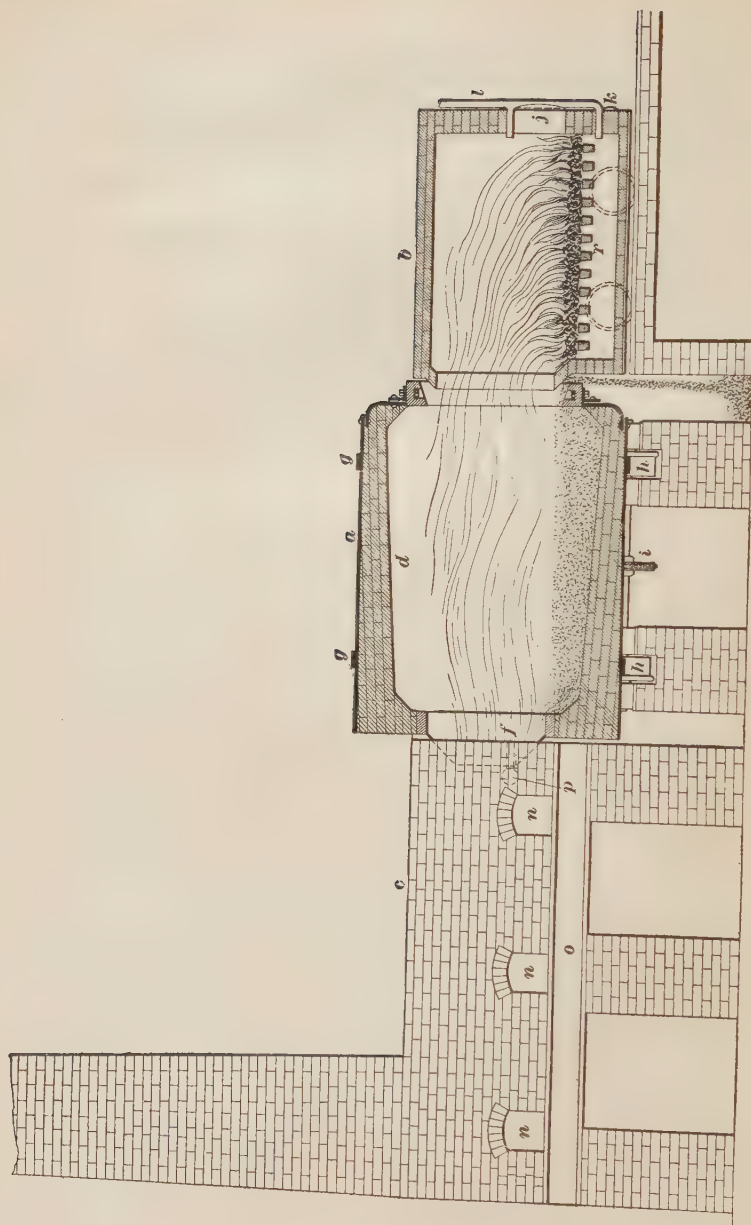


FIG. 22

as in the soda process owing to the greater use of the indirect cooking in sulphate mills. These evaporators have the advantage that they will handle thick, gummy liquors that would clog tube evaporators.

102. Rotary Furnaces.—The liquor from the evaporators is sufficiently concentrated so that it will support its own combustion when introduced into the rotary furnace. A storage tank heated with a steam coil should be placed in the system between the evaporators and the rotary furnace, and a by-pass should be installed so the liquor can go direct from the evaporators to the rotary when circumstances permit. In this way the maximum amount of heat can be conserved. The furnace consists of two parts, the rotating part *a* and the stationary part *b*. The stationary part is a firebox, constructed either of steel plate lined with firebrick, or of firebrick held in place by iron bands. It is fitted with a grate *r* for burning coal or wood, or with equipment for burning gas or oil. It opens into the rotating part of the furnace. The rotating part is a cylindrical steel-plate shell, lined with firebrick. It is usually from 8 to 9 feet in diameter, unlined, and from 14 to 30 feet long. It leads into a flue *c* provided with clean-out doors *n*. The lining decreases the diameter about 1 foot. It requires about 1 pound of coal burned in the firebox to produce 6 pounds of ash in a 14-foot rotary, while 1 pound of coal will produce 12 pounds of ash in a 30-foot rotary. When burning liquor from cooking of hardwood, 70 pounds of ash per square foot of burning surface per 24 hours is considered good, and this is increased about 10 per cent. for more resinous woods such as spruce, pine, poplar, etc. However, the larger rotaries cause more loss of soda through the stack owing to the increased draft required. The furnace is caused to rotate slowly. This is usually accomplished by iron rings *g* which rest on flanged wheels *h*, the axles of these wheels resting in journals supported by masonry. The rotation is accomplished with a worm-drive and a gear and pinion *i*. The liquor enters by a line which passes through the wall of the flue at *p* and is run into the end of the rotating cylinder

farthest from the firebox at *f* and gradually runs forwards burning as it moves, and the black ash falls out of the cylinder through an opening located just back of the firebox. The heat produced in this furnace is generally utilized, either in the disk evaporators or for operating a boiler which supplies steam to the multiple-effect evaporators. The black ash used to be conveyed, either by trucks or by a conveyer to the leaching and causticizing department, but is now more usually discharged into a tank situated in a pit under the discharge of the rotary. The liquor from this tank goes to the alkali room where it is used in making up the soda liquor for charging the digesters as already described.

103. Calculation of Recovery.—Recovered black ash contains from 43 to 48 per cent. of Na_2O . When figuring recovery, in some mills the black ash made is weighed and the recovery calculated from the amounts of each ash used in making up the liquor. The recovery is usually calculated by multiplying the amount of 48-per-cent. soda necessary for one digester by the number of digesters put on, subtracting the amount of soda used in the alkali room (after calculating the latter to 48-per-cent. Na_2O), and dividing the remainder by the total amount used, as just calculated. The amount of black ash recovered varies from 75 to 90 per cent. in the different mills.

The sources of loss of soda through the mill are: (1) retention in the dump of lime sludge from causticizing pans; (2) blowing of the digester; (3) imperfect washing of stock in the wash pans; (4) dust carried up the chimneys of the rotaries; (5) formation of silicate of soda due to silica present in the lime; and (6) leaching the black ash, under which source will also come loss due to imperfect burning, as this is very frequently the cause of a considerable loss of soda. The caustic liquor used in boiling straw and esparto is also recovered by a process similar to the one just described.

104. Loss of Soda Up Stack.—One of the biggest problems confronting the soda-pulp mill superintendent today is that of recovering the soda passing out of the rotary stack.

Some have tried to do so by a wet method, by spraying the stack with weak liquor or water, but so many products of combustion and destructive distillation are reclaimed with the ash, that it forms a dirty conglomerate mass, difficult to handle. It is too dirty to send to the leachers, not strong enough to burn without evaporation, and contains too much suspended solid matter to send to the evaporators. The washing has been accomplished very efficiently; but, as stated, it has been a very difficult matter to prepare the resultant liquor for use.

Attempts have been made to separate the solids from the liquor by passing it through a plate and frame filter press, but this process is very slow and unprofitable.

105. There have been several methods suggested for collecting the dust in a dry way, the most feasible of which appears to be the Cottrell electrical precipitation process. By this method the dry dust is precipitated electrically, while the moisture and gases pass out, owing to the high temperature in the stack.

This is an expensive installation, as the units are large, requiring 360 collecting electrode pipes, 15 feet long and 8 inches in diameter to handle 50,000 cubic feet of gas per minute. Moreover, special electrical-generating, transforming, and controlling equipment is required. The operating expenses, including interest, depreciation, royalty, power, labor, etc., would be a little less than \$20,000 a year on such an installation. If, by draft control, it is impossible to keep the stack losses below 7,000 pounds of soda a day in a rotary department sending 50,000 cubic feet of gas per minute up the stack, it is advisable to use this or some other dry dust-saving system.

106. While it may not seem advisable to install an expensive system to reclaim the ash, this important point in the recovery department should not be overlooked. It should be under efficient control at all times, and the speed of the flow of gas so regulated as to produce the best burning results, with the smallest possible loss of soda. The velocity of the gas entering the nozzle of the dust collector should be the

same as that in the flue. In order to measure the gas at the proper velocity it is pulled through a water-filled gas meter by means of a steam or an air siphon. The dust can be caught in paper thimbles when the temperature is under 450° F. If the temperature exceeds this, alundum thimbles should be used.

By tests made in the foregoing manner it has been found that 14-foot rotaries lose from 450 to 1,000 lb., 16-foot rotaries from 650 to 1,300 lb., and 30-foot rotaries from 2,500 to 4,000 lb. of soda up the stack in 24 hours.

Many varieties of dust-collecting chambers have been constructed between the rotary and the stack, some of which are fairly efficient; but, as a general rule, the ash collected represents only a small percentage of the amount of ash passing up the stack.

SULPHATE PROCESS

107. Nature of Sulphate Process.—The sulphate process is a modification of the soda process in which sulphate of soda is used to replace soda ash. This process was invented by Dahl in Europe about 40 years ago. The first introduction of the sulphate process in America was in Canada in 1907, since which time the industry has developed rapidly, the United States and Canada today producing about 2,500 tons per day. It was originally used for making a pulp, which was subsequently bleached, yielding a very fine grade of stock from which excellent soft pliable papers, suitable for book and other purposes could be made. A considerable amount of sulphate pulp is still prepared which is subsequently bleached, especially in Europe, but the greatest application of the sulphate process today is in making what are known as *kraft papers*. Kraft paper is an exceedingly strong, tough paper, ideally suited for wrappings and bags. It is not bleached, having a natural brown color which is characteristic of this variety of paper. As there is a great demand for this product and as a number of woods can be reduced to pulp by this process much more economically than by any other, manufacture of kraft paper by the sulphate process has assumed very large proportions in the paper industry.

Originally kraft paper was made by only partly digesting the pulp and subsequently completing the process by mechanical means such as grinding in a *kollergang* or edge runner. This procedure required a great deal of power and also gave a very limited output as the capacity of the *kollergang* was comparatively small. In the modern production of kraft pulp, the cooking is more thorough, and the subsequent disintegration of the pulp is accomplished in the beaters and jordans. The sulphate process costs more to operate than the soda process, especially as concerns the recovery system.

108. Cooking Liquor.—Although the process is known as the sulphate process because sodium sulphate is used in making up the liquor, a number of other compounds of soda are present in the liquor as used. The liquor as actually used contains in solution sodium hydroxide, $NaOH$, sodium sulphide, Na_2S , sodium carbonate, Na_2CO_3 , and sodium sulphate, Na_2SO_4 . The actual digestion of the pulp is mainly effected by the sodium sulphide in the liquor, and it has been suggested that *sulphide process* would be a more correct name than *sulphate process* for this method of making pulp. A high percentage of sulphate or carbonate in the cooking liquor is undesirable.

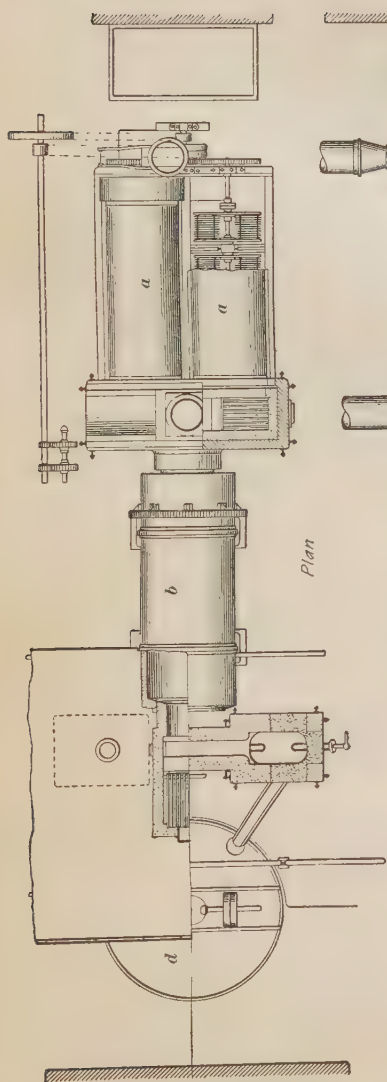
109. Equipment.—The digesters used for the sulphate process are practically identical with those previously described in connection with the soda process. They must be well insulated to avoid heat losses and excessive condensation. Just as in the soda process, no special lining is required such as is used in the sulphite process, because the cooking liquor does not attack the metal of the digester. Direct cooking, such as is used in the sulphite process, is extensively employed. However, various systems of indirect cooking have recently been applied successfully, the digesters being equipped with a system of forced circulation.

110. Details of Sulphate Process.—The wood is prepared for the sulphate process exactly as for the soda process. It is chipped just as for the soda or sulphite process and the chips are stored in bins from which they are run into the

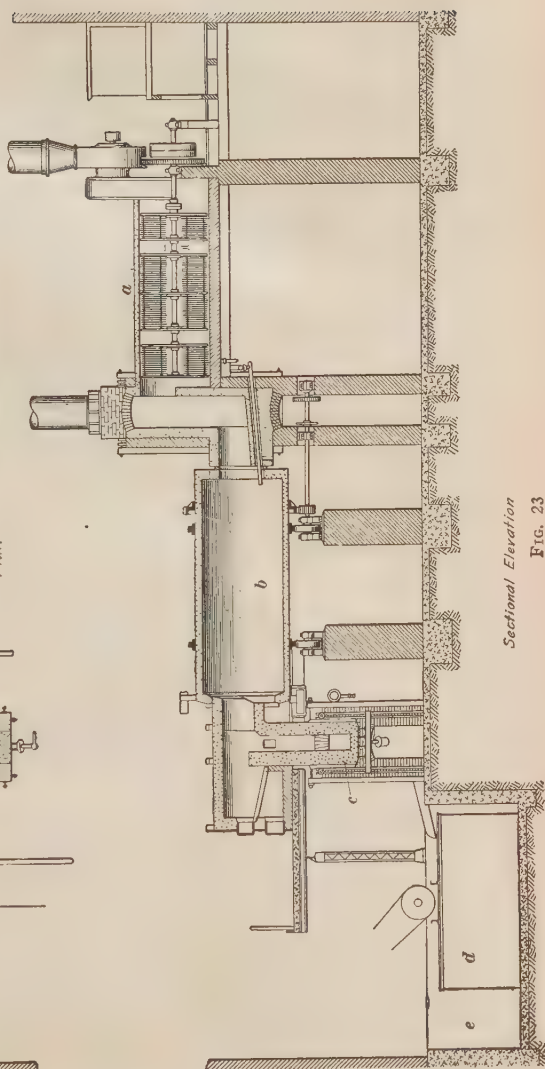
digesters. After the required amount of chips has been placed in the digester, the liquor is run in and the digester closed. The pressure is brought up to 20 or 25 pounds, at which point the cold air present in the charge is relieved. Immediately thereafter, the pressure is brought up to maximum, which is usually about 100 pounds, and no relieving is necessary from that point on as, unlike the sulphite cook, no gas is formed during the cooking. There is no top pressure and the temperature and pressure will correspond right up to the end of the cook.

111. The duration of the cook depends on the quality of pulp to be produced and also on the percentage of moisture present in the wood, and other factors. From $2\frac{1}{2}$ to 6 hours, depending on conditions, is usual; and short cooks, say $3\frac{1}{2}$ hours, predominate. When the cook is completed, the pulp is blown into the blow pit, just as in the sulphite process. In making the sulphate pulp it is customary to blow the digesters at a somewhat lower pressure than is usually the case with sulphite digesters. This is done by relieving the pressure just preliminary to the blow. It is claimed that blowing at low pressure will yield a better quality of pulp in addition to making the washing of the pulp in the blow pit an easier matter.

112. The steam blown off is passed through a separator which eliminates liquor and turpentine which is always formed in connection with this process. The steam is used to heat some of the water used in washing the pulp. A very thorough washing is necessary for this class of pulp, not only to give a pure stock, but also to recover the highest possible percentage of chemicals used in the process. As a general rule the pulp is washed in a series of diffusing tanks. The diffusing tanks are closed, as the vapors given off in the sulphate process are malodorous and noxious. In the first of these tanks it is washed with waste liquor and in each of the succeeding ones with more and more dilute liquor until it is finally washed with pure water. By this process, the percentage of soda in the liquor is systematically concentrated.



Plan



Sectional Elevation
FIG. 23

113. The waste liquors all go to a recovery system, such as is illustrated in Fig. 23, and the efficient operation of this recovery system is the most important detail in connection with the successful operation of a sulphate mill. The recovery system consists of evaporators *a* and rotary furnaces *b* quite similar to those already described in connection with the soda process. Where the indirect cook is used, multiple-effect evaporators are sometimes dispensed with, the liquor being sufficiently concentrated to go direct to the disk evaporators.

However, the latter stages of the recovery are somewhat different owing to the fact that sulphate of soda is substituted for soda ash in the process. The black ash from the rotary furnace is mixed with sulphate of soda and sawdust and thrown into a smelting furnace *c* lined with soapstone (alberene stone), or refractory brick. Air is forced into the furnace and the black ash is burned, during which operation part of the sulphate of soda is reduced to sulphide of soda. Some carbonate of soda is also produced and some of the sulphate of soda passes through this process unaltered. The hot gases from this furnace are used to heat the rotary furnace in which the final concentration of the waste liquor has previously taken place. The fused alkali from the smelting furnace runs into a dissolving tank *d* set in a concrete pit *e* and fitted with an agitator where it is dissolved with the proper quantity of water or weak liquor from the washing of the lime mud in the causticizing department. It is then pumped to the causticizing system. The causticizing system and the general arrangement for preparing the liquor for charging the digester are not materially different from those used for the soda process.

114. The fresh liquor from the causticizing department, usually known as *white liquor*, is generally made stronger than is intended to be used in the digesters, and is subsequently diluted with black liquor, thus making up the volume and concentration required for the digester. This is generally done with tanks provided with floats. The tanks have previously been calibrated; that is, the operator knows how many gallons are represented by an inch of height on the tank. The com-

position of the cooking liquor depends on many factors, such as the nature of the wood, the duration of the cook, etc., but in general it should analyze about 40 to 65 grams per liter of caustic soda and about 15 to 40 grams per liter of sodium sulphide. The total amount of mixed caustic soda and sodium sulphide will be about 16 to 23 pounds per 100 pounds of wood to be pulped, on a bone-dry basis. The sulphate and the carbonate should be kept low in the cooking liquor, as neither of these chemicals has any active part in the pulping process. The sulphate will be high if the reduction in the smelting furnace is not properly conducted. Incomplete causticization is the chief cause of an excess of carbonate. Even if the reduction should be complete in the smelting furnace, there will be some sulphate in the liquor, as sodium sulphide will always gradually oxidize to sulphate in solution. In actual practice the liquor contains small amounts of many other compounds of sulphur and soda.

MANUFACTURE OF PAPER

(PART 2)

BLEACHING AND BEATING

BLEACHING

1. Bleaching Fibers.—After washing, the various fibers produced by any of the paper-making processes are always found to be more or less colored, owing to the fact that a portion of the noncellulose constituents survives. The next operation is to remove as much as possible of this coloring matter and thereby produce pure-white fiber for the manufacture of the best grades of paper. This is accomplished by the bleaching process.

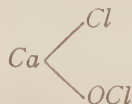
The bleaching of rag stock is a comparatively simple matter owing to the comparative freedom of such stock from colored impurities which have to be eliminated by the bleaching agent. It should be borne in mind that most rag stock of the better grade is made from material which has already been submitted to bleaching in the processes of textile manufacturing, and any coloring matter which may be present is in the form of dyes which have been added by the textile manufacturer or finisher, and which are relatively easy to remove when compared with the coloring materials embodied in wood pulp which are an integral part of the fiber itself.

Wood pulp, no matter how carefully made, and whether produced by the sulphite or soda process, always has associated with the cellulose a portion of the lignin or incrusting matter ordinarily present in the raw fiber, and this lignin carries

with it certain colored bodies of highly complex chemical composition. These colored impurities cannot be removed by any amount of washing or mechanical treatment. They are united chemically with the fiber or cellulose and a chemical process is necessary for their removal.

2. In addition to the colored materials that are ordinarily present in the fiber, other dark-colored substances are produced during the process of digesting the pulp, by the chemical action of the acid or alkaline liquids on the various complex substances contained in natural wood. Wood pulps and pulps made from esparto, straw, jute, etc., require, as already pointed out, a much more drastic bleaching than rag pulp, resulting in a much larger consumption of the chemical used for bleaching purposes and a much greater proportional loss in weight through the bleaching process. The object of all successful bleaching practice in the paper industry is to bleach the pulp thoroughly so as to turn out a product of maximum whiteness and purity, which will remain white indefinitely, and, at the same time, not to impair the strength and natural properties of the fiber, not to cause too much shrinkage in weight and volume, and not to have an excessive consumption of the bleaching agent.

3. **Chemistry of Bleaching.**—Bleaching is simply an oxidizing action, and although there are other agents, the principal ones used in bleaching are chlorine or compounds of chlorine. Of these compounds of chlorine, bleaching powder, which is made by passing chlorine gas through slaked lime, is the one most used in the paper industry. The formula of bleaching powder is generally accepted as



It is also generally accepted that the bleaching action is due to the liberated chlorine combining with the hydrogen of the water, according to the equation $H_2O + Cl_2 = 2HCl + O$, liberating nascent oxygen, which does the bleaching.

Bleaching powder readily absorbs moisture from the air, and for this reason must be kept in covered drums or other vessels that will exclude air. It is bought and sold on the basis of the amount of available chlorine present in the bleaching powder. Good commercial bleaching powder, or *chloride of lime*, as it is frequently called, should contain from 35 to 37 per cent. available chlorine.

4. Bleaching powder is usually shipped in steel drums or wooden barrels. The steel drums weigh from 100 to 800 pounds, including the weight of the drum, and the wooden barrels usually weigh from 350 to 415 pounds, including the weight of the barrels. Where it is necessary to use this material in small quantities, it is usually purchased in 5- or 10-pound

TABLE I
RELATION OF FRENCH DEGREES TO PERCENTAGE OF
AVAILABLE CHLORINE

French Degrees	Percentage Available Chlorine	French Degrees	Percentage Available Chlorine
65	20.65	100	31.80
70	22.24	105	33.36
75	23.83	110	34.95
80	25.42	115	36.54
85	27.01	120	38.13
90	28.60	125	39.72
95	30.21	130	41.34

cans, and this is frequently a convenient way of buying and storing the material as the slightly increased cost on account of the containers will be more than offset by the prevention of deterioration in the material.

In Europe bleaching powder is frequently sold on a degree basis, the degrees representing the volume of chlorine which will be liberated from 1 kilogram of the bleaching powder at standard temperature and pressure. The relation of French degrees to available chlorine is shown in Table I.

5. Bleaching powder requires great care in shipment and storage. If the powder is allowed to become wet, or even damp, it will deteriorate rapidly and lose a large percentage of its available chlorine. In case the powder should become actually wet, as might happen in a leaky ship or a bad car exposed to the weather, the decomposition may be so rapid as to cause explosions.

Griffin and Little mention a series of experiments carried out by Pattinson regarding the rate at which bleaching powder

TABLE II
COMPOSITION OF BLEACHING POWDER

	January 29, 1885			January 5, 1886		
	A	B	C	A	B	C
Available chlorine . . .	37.00	38.30	36.00	38.80	35.10	32.90
Chlorine as chloride . .	.35	.59	.32	2.44	2.42	1.97
Chlorine as chlorate . .	.25	.08	.26	.00	.00	.00
Lime	44.49	43.34	44.66	43.57	42.64	43.65
Magnesia40	.34	.43	.31	.36	.38
Silicious matter40	.30	.50	.50	.40	.50
Carbonic acid18	.30	.48	.80	1.48	1.34
Alumina, peroxide iron, oxide manganese48	.45	.35	.40	.40	.37
Water and loss	16.45	16.33	17.00	18.18	17.20	18.89
	100.00	100.00	100.00	100.00	100.00	100.00
Total chlorine	37.60	38.97	36.58	41.24	37.52	34.87

deteriorated in storage. Pattinson took three 600-pound barrels of bleaching powder, and from each of the barrels he filled twelve bottles with powder. The barrels were sealed and both the barrels and bottles were stored in a cellar. A maximum and minimum thermometer was placed near them, and a careful record of the temperature made for each working day of the year. The record shows the temperature to have been uniform and comparatively low during the entire year, the highest being 62° F., and the lowest 38° F.

One bottle from each of the three sets of twelve was opened and tested each month, and a sample from each of the three casks was also withdrawn and tested. The results of the experiment show a gradual and regular loss of available chlorine during the time over which the tests were made. The average loss in the barrels was about one-third of 1 per cent. greater than in the bottles, and the barrels were not necessarily air-tight. A complete analysis of each of the barrel samples was made at the beginning and also at the end of the experiment. These analyses are given in Table II.

6. The small quantity of chlorine found as chlorate at the beginning of the experiments ceased to exist in this combination at the end, and from tests made it was found that all the chlorate had disappeared about 4 months after the barrels were filled. The amount of chlorine existing as chloride had slightly increased. It is not often that bleaching powder can be stored where a temperature as low as 60° F. can be maintained for any length of time, especially in the summer months when, as previous experiments have indicated, the greatest loss of available chlorine takes place.

Bleaching powder should be stored in as dry a place as possible, or better, in a place that is both dry and cool; and if any of the containers are damaged, they should be the first ones selected for use.

7. According to Griffin and Little, the rate at which the bleaching powder deteriorates is influenced by the kind of container in which it is packed. Soft woods are considerably affected by the action of the powder, and shrink badly when exposed to the sun. If such containers are subsequently exposed to rain, water readily finds its way into the barrels. Ash and other hardwoods can be used for such barrels, but the best barrels are built of oak staves 1 inch in thickness.

The leading companies manufacturing bleaching powder have given very careful attention to the containers in which this material is shipped, and if it is purchased from a reliable concern, little trouble will probably be experienced with deterioration in shipment or storage.

8. Preparation of Solution of Bleaching Powder.

The tanks used in preparing the solution of bleaching powder are generally made of iron and painted with red lead ground in oil, and are provided with agitators and siphon pipes. They vary in size and hold from 1,000 to 2,500 gallons. The most convenient size is one in which either one or two whole casks of bleaching powder can be used and the bleach made up to the required strength, thus avoiding the necessity of splitting casks. The temperature of the water used in mixing the bleach should not exceed 70° F., and it is better, provided the bleach settles well, to keep the water as near 60° F. as possible.

9. The tank is filled about two-thirds full of water, the agitators started, and the bleaching powder dumped in. The agitation is continued until all the lumps are well broken up, after which it is stopped and the bleach allowed to settle. The agitation in mixing the powder should not be too prolonged, as it will not settle so well. Agitating from 15 to 20 minutes is sufficient for a strong bleach. The washes are agitated while filling and are stopped when full. In some mills, just enough bleach is used to make the liquor up to the required strength, the washes being used for making up other tanks and washes, and the final wash being made with water. The method most generally adopted is to make the bleach up stronger than desired and to mix in a tank in the cellar with washes of other tanks until the required strength is reached. Sufficient washes should be made until practically all the chlorine has been washed out of the sludge, allowing the bleach to settle after each wash. The loss in sludge should not exceed 1 per cent. of the total amount of bleach used in making the solution. The balance of these washes, after the strong bleach is diluted to the required strength, is run into a separate tank and used in place of water for making strong bleach. The strong-bleach solution is usually made up to about 7½° to 8° Baumé, and after dilution it tests from 3½° to 4½° Baumé, at which strength it is used in the bleachers.

10. A hydrometer is generally used for testing the strength of the bleaching liquor. This is a very inaccurate

method of testing this liquor because there is not necessarily any definite relation between the density of the solution and the amount of available chlorine present. If the mill has adequate laboratory facilities it would be much better to take samples at regular intervals and have these sent to the laboratory for a determination of the available chlorine present. However, the hydrometer test is better than nothing, and in many mills is the only practical method that can be used. It may be accepted as a rough rule that 1° Baumé on the hydrometer averages about .47 per cent. available chlorine in the solution.

The percentage of available chlorine is not the only factor to consider when purchasing bleach, as the settling quality figures to a considerable extent. It is advisable that only clear liquor be used in bleaching, and for this reason a bleach that is high in available chlorine, but a poor settler, is of no more value to a concern that has a limited capacity for making bleach than a bleach that has less available chlorine, but settles well.

The sludge from bleaching powder, consisting principally of calcium hydroxide and calcium carbonate, is washed out into the sewer after the last wash has been run off. Whereas bleaching was formerly carried out in simple tanks provided with more or less crude agitators, at the present time numerous highly efficient special forms of bleaching equipment are on the market, all of which are designed with the idea of making the process as largely automatic as possible.

11. Bleaching of Rags.—Rag pulp is frequently bleached in a *hollander*, or washer in which the boiled rags are given the preliminary treatment which converts the stock into what is known as *half-stuff*. As a rule, in bleaching this kind of stock, no special bleaching equipment is provided, the bleaching agent being added to the hollander towards the final stages of the operation and washing being continued sufficiently long after the bleaching effect has been accomplished to wash out the impurities and the surplus bleach. Hollanders used for this purpose are often called *potchers*.

The bleaching of rags is better accomplished by adding either hydrochloric acid or sulphuric acid during the process

of bleaching. The acid should be very much diluted when added, but the addition should not be made until the bleach has been acting on the pulp for some time. When acid is added to accelerate the bleaching action, the process is termed *acid bleaching*. Acetic acid has been used extensively to assist the action of bleach liquor in the bleaching of rags, which only requires the addition of a small quantity, from the fact that during the operation the acid is regenerated according to the following equations:



The amount of bleach necessary to produce a good color depends on the thoroughness of previous treatment, but may be given as from 2 to 5 pounds of bleaching powder for every 100 pounds of pulp.

12. Bleaching of Esparto.—Esparto fiber is very often bleached in the washing and beating engine, where it is subjected to an acid bleaching. The required quantity of bleach liquor is added, and after mixing for about half an hour, the highly diluted acid is added (using about 6 ounces of acid to 100 pounds of fiber) and the bleaching continued until a good color is produced. The liquor used tests about 4° Baumé at 60° F. Esparto fiber is also bleached in large potchers made of brick and which are lined with cement, the agitation being accomplished by means of large revolving paddles made either of wood or iron, preferably the latter. From 10 to 15 pounds of bleaching powder is required to bring 100 pounds of esparto to a good color.

13. Bleaching of Straw.—Straw fiber is bleached by methods similar to those used for bleaching esparto. The amount of bleach required is from 8 to 12 pounds for each 100 pounds of pulp.

14. Bleaching of Jute and Manila.—Jute and manila are usually bleached in the washing engine, and chloride of lime, when used, is added in a very weak solution, which

bleaches the fiber to a cream color, oxidizing it to some extent. Strong bleach should not be used, as it will oxidize the fiber. It is better to use a weak solution of sodium hypochlorite in the bleaching of jute, as a solution of this kind will prevent the formation of the oxidized compound. As it is practically impossible to bleach jute fibers to white, they are generally used for papers that do not require a high color. From 9 to 10 pounds of bleaching powder is required for each 100 pounds of pulp.

15. Bleaching of Ground Wood.—Owing to the fact that ground wood contains nearly all the intercellular constituents of the wood, which have to be removed by the bleach before any action takes place on the coloring matter of the fiber, it cannot be economically bleached, and it is therefore used for papers where perfect whiteness is not a consideration.

16. Bleaching of Chemical Wood Fiber.—There are many forms of bleaching equipment for bleaching chemical wood fiber made by the sulphite, soda, and sulphate processes. Some of these very much resemble a beating engine (in fact, some paper makers bleach their stock in the beaters), others are large cylindrical wooden tanks provided with an agitator, which consists of a central rod with paddles attached at different heights while still others are large, open, tile-lined vats made of brick, having wings attached to a revolving horizontal shaft so as to keep the stock agitated during the bleaching operation. It has been found by practice that better results can be obtained by bleaching the stock in open bleachers. All forms of bleachers have a steam line running into them, by means of which the stock can be heated during the operation. The heating must be done very cautiously, as there is great danger of oxidizing the fiber if heated too highly; there is also danger of heating it too highly in one spot (where the steam enters), with the same result. Better results can be obtained by heating the stock to the required temperature before adding the bleaching liquor. It is advisable not to exceed a temperature of 115° F. If this temperature is exceeded, there will be trouble from time to time with oxidized fiber.

17. At times, owing to insufficient treatment in previous operations, it is difficult to bring a bleacher of stock up to color. In such a case, the action can be greatly assisted by washing out the products of the bleaching action, treating with a weak solution of alkali, and washing again. After this treatment, the most refractory pulp can usually be brought up to color by again treating with bleaching solution. In bleaching sulphite pulp, the fact that from 14 to 22 per cent. of bleaching powder is required indicates that the powder has to perform other actions besides bleaching. The amount of bleaching powder required increases with the amount of incrusting matter left in the pulp. Using the soda solution just mentioned before bleaching will greatly reduce the expense of bleaching.

18. In bleaching sulphite pulp, some paper makers warm the pulp in a 13-per-cent. solution of bleaching powder, and after 1 hour's time add a 2.5-per-cent. solution of sulphuric acid. The pulp is then washed for $2\frac{1}{2}$ hours and rebleached with a 2-per-cent. solution of bleaching powder, finally adding a $\frac{1}{2}$ -per-cent. solution of sulphuric acid.

In bleaching soda or sulphite fiber, the strength of the bleach solution used is generally from $3\frac{1}{2}^{\circ}$ to 4° Baumé at 60° F. (about $\frac{1}{2}$ pound of bleaching powder to a gallon). It is advisable to keep the solution as regular as possible in order that the results obtained may be uniform. The bleaching of wood fiber requires from 12 to 25 pounds of bleaching powder for each 100 pounds of pulp, depending on the wood used, the process by which the fiber was isolated, etc. After bleaching any fiber, it must be well washed in order to remove the excess of bleach and soluble by-products. This is done in some mills by means of a drum washer in the potchers; in others, by the same operation in the beaters; and in still others, the bleached stock is pumped with a large quantity of fresh water to large drainers, or chests, having perforated bottoms and allowed to stand until it drains down solid, after which a large amount of water is added and the stock is pumped to the beaters, mixers, or pulp-machine stuff chest.

19. In order to get good results from bleaching, the stock should be well agitated. The question of agitation has caused considerable experimenting to be done by the management of the various paper mills, and as a result, some mills have adopted a system of continuous circulation. In this system, the stock, after the bleach has been added, is taken to a battery or a set of bleachers, through which it is made to pass, being pumped from the bottom of one bleacher to the top and opposite end of the other, and then from the bottom of the second bleacher, on the opposite side from where it entered,

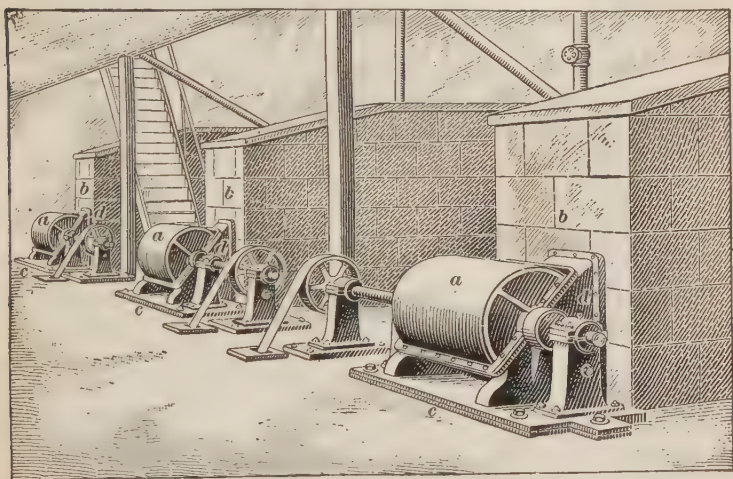


FIG. 1

to the top of the third, and so on through a series of about six bleachers, all of which are furnished with agitators.

20. **Bellmer Bleaching Process.**—Another very successful system is the Bellmer bleaching process, an installation of which is illustrated in Fig. 1. Propellers *a* are erected outside the tubs of the bleaching engines *b* on a cast-iron foundation plate *c*. They are connected with the engine tub by means of flanges and enclosed funnels *d* projecting in the walls of the tub. The propellers are driven by a belt running from above or below the floor. When electric motors are

used the propeller axle is connected directly to the motor. The frame can easily be opened at any time by removing the top. The tub *b* is generally made of concrete or brick and is lined inside with tile, which require little cleaning and protect the inside of the tub from the injurious action of the bleaching fluid. Moreover, the tile lining reduces the friction of the material against the sides and bottom and helps to keep the bleach liquor clean. The tub has no dead points and is designed with enough bottom fall to insure perfect circulation. The dimensions of the tubs can be altered to suit local conditions and they can be installed singly or in batteries.

21. Double-acting propellers with three running channels are best, but single-acting propellers can be installed when desired for installations handling less than 8,000 pounds. The advantages of this system are as follows: The propeller admits the thickest material and will move the contents rapidly enough to be economical; the mixing of the bleaching liquor with the stock is thorough; formation of scum and knots is avoided; no separate stuff pump is required; various kinds of stock can be mixed while the bleaching operation is going on.

As nearly all bleach solutions contain some chlorate, which is inactive as a bleaching agent, it is generally advisable to add a small quantity of sulphuric acid. This acid should be added when the available chlorine has just about been exhausted, or when the stock shows a pale-blue color with iodic starch. Also, time should be allowed for its action before sulphurous acid or bisulphide liquor, used as a antichlor, is applied. The sulphuric acid should be very much diluted before it is added (about 1 quart of acid to a barrel of water), and should be run in very slowly. The adding of sulphuric acid will liberate the chlorine from the chlorate and will give the liberated chlorine an opportunity to assist in bleaching the stock.

In cases where the bleaching capacity of the plant is insufficient, it is advisable to add a little sulphuric acid to hasten the bleaching action. In this way it is possible to bleach more

economically, and, in addition, the washing of good bleach solution from the stock is avoided. Great care, however, must be exercised in thus forcing the bleaching.

22. Bleach Consumption.—The amount of bleach required for any particular lot of stock depends, as previously explained, on many factors. It is the general opinion that the majority of mills use more bleach than is necessary. There is a tendency to use bleach liberally, getting rid of any excess of bleach with antichlor. This is wasteful and expensive.

The figures given in Table III are approximate for the amount of bleach required for 100 lb. of different fibers:

TABLE III
BLEACH REQUIRED FOR A HUNDRED POUNDS OF
DIFFERENT FIBERS

Fibers	Amount Required Pounds
Rags	2 to 5
Straw	7 to 10
Esparto	10 to 15
Soda (poplar)	12 to 15
Soda (spruce)	18 to 25
Sulphite (poplar)	14 to 20
Sulphite (spruce)	15 to 25
Jute	10 to 20

23. Antichlors.—It is generally customary to use some form of antichlor to neutralize the last traces of bleach after the stock has come up to the required color. Sulphite and hyposulphite of soda are used to a great extent for this purpose. Sulphurous acid is also used to some extent. This acid removes the slight yellow tint left in the pulp after bleaching, bringing it to a fine white. On exposure to the atmosphere for any length of time, this yellow tint will appear again, because the coloring matter that was temporarily removed by the reducing action of the sulphurous acid will

again become oxidized. When treated with an antichlor, the pulp must be rewashed.

24. Bluing.—A small amount of blue (ultramarine or some other blue) is frequently added to offset any slight yellowness that may be left after bleaching. Sometimes this is done with pulp that is to be offered for sale to cover up imperfect bleaching. The addition of even very little blue soon becomes apparent to the eye of the expert. It can best be detected by rolling a lap of the pulp into a tube and looking into it by a clear north light, or by looking into a folded lap as into the pages of a half-open book.

25. Electrolytic Process for Making Bleaching Liquor.—Methods of preparing bleach liquor by the electrolysis of common salt have been developed to a high degree of efficiency, and as a result of these experiments such a degree of proficiency has been gained that electrolytic bleach plants have been installed in a large number of pulp and paper mills. The process of preparing electrolytic bleaching liquor is based on certain well-known principles of electrochemistry, a general idea of which is here given.

A metallic conductor does not suffer any apparent change when a current of electricity passes through it, but various magnetic and heating effects are produced. Some liquids, as well as solids, are good insulators, while others conduct electricity, and are termed *electrolytes*. The latter suffer decomposition in proportion to the amount of current passing through them. The points at which the current enters and leaves the electrolyte are called the poles. The entering pole is designated as the *anode*, and the pole at which the current leaves is called the *cathode*. The products of decomposition of the liquids are observed at the poles, and are called *ions*; that liberated at the anode is termed the *anion*, and that liberated at the cathode, the *cation*. In the process of decomposition of fused common salt, chlorine is given off at the anode and sodium at the cathode. When a solution of common salt is used, a secondary reaction takes place, due to the contact of the liberated ions, and there is a tendency toward the forma-

tion of sodium hypochlorite, which remains in solution, and hydrogen, which escapes at the cathode. There is also a decomposition of the water itself into hydrogen and oxygen, and the oxygen that is liberated at the anode will attack the material of which it is made. If the anode is of carbon, it will be destroyed in a short time. The aim of the inventor is to procure an anode that will resist the action of the products of electrolysis. Platinum is best in this respect, but as it is very expensive, different forms of carbon are used in cells in practical use. It is also advantageous to use as little water as possible hence, nearly all inventors use a saturated brine solution. The quantity of electrolyte decomposed by the passage through it of a given quantity of electricity is always the same.

26. That which causes electricity to flow from a point of high potential to a point of low potential is called the electromotive force (E. M. F.), the unit of which is the *volt*. The unit of quantity of current is the *coulomb*; the unit of rate of flow, which is 1 coulomb per second, is called the *ampere*; and the unit of resistance to the flow is the *ohm*. An electromotive force of 1 volt will send a current of 1 ampere through a resistance of 1 ohm. A current of 1 ampere, theoretically, yields 1.34 grams of chlorine and 1.51 grams of caustic soda per hour. Owing to complications due to secondary reactions, the yield in practice is only about 1 gram of chlorine per ampere per hour. The power of a current in doing work is measured in units called *watts*. A current of 1 ampere, under an electromotive force of 1 volt, has an energy of 1 watt. One horsepower equals 746 watts.

Two pounds of coal is converted into 1 horsepower of mechanical energy, which, as stated, is equivalent to 746 watts. This is converted through the dynamo (with customary loss) into about 650 watts, which is the efficiency of the dynamo for each horsepower.

An electromotive force of from 3 to 5 volts is required between the terminals, and since the power of a circuit in watts is equal to the number of amperes flowing multiplied

by the electromotive force in volts, to produce 1,000 grams of chlorine it will require (assume the electromotive force to be 4 volts) 4,000 watts, or about 6 horsepower per hour, which means the consumption of 12 pounds of coal.

27. A great difficulty experienced in the process thus far set forth is in getting the caustic-soda solution free from salt, as a diaphragm is required between the anode and the cathode that will furnish as little resistance as possible and at the same time prevent the passage of brine solution through it. Several different materials are in use for diaphragms, such

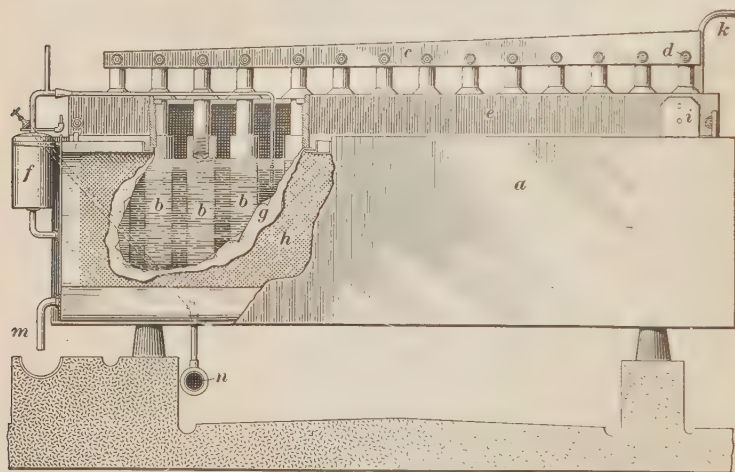


FIG. 2

as unglazed earthenware, asbestos, etc. A great many processes have been proposed for the production of chlorine and caustic soda by electrolysis, all of which have the same fundamental principles underlying them, namely, working with a saturated brine solution, having a diaphragm between the poles, and separating the chlorine from the soda. There has, however, been considerable difference in the designs of the apparatus.

28. Electrolytic Cells for Preparation of Bleach. The Nelson cell, which is typical of modern equipment,

consists essentially of a steel tank containing a perforated-steel cathode sheet and a number of graphite anodes. Supported by the steel cathode sheet is an asbestos diaphragm. Fig. 2 shows a side view of the cell, with the side of the steel tank *a* broken out to display the interior construction. The graphite anodes *b* are seen suspended in the anode compartment, surrounded by brine. The graphite anodes are connected together by a tapering copper bus-bar *c*, and are suspended in the anode compartment by pins *d* resting on the top of the slate gas dome *e*, which serves to carry away the chlorine evolved. The brine solution enters through the pipe *n* and is kept at constant level by means of the automatic brine feed *f*, attached to the left-hand end of the cell. Direct current flows through the anodes, electrolyte, and asbestos diaphragm *g* to the perforated-steel cathode sheet *h*. From this cathode the electric current flows through two copper ears *i* attached to each side of the cathode sheet, and through copper connectors to the next cell. Chlorine is set free at the anodes, rises through the electrolyte and passes into the slate gas dome *e*, from which it is discharged through a glass pipe *k* by a fan or pump. The electrolyte percolates through the asbestos diaphragm *g* to the perforated-steel cathode sheet *h*, where sodium is formed, which reacts immediately with the water of the brine, forming caustic soda and hydrogen. The hydrogen evolved in the cathode compartment either escapes to the air through outlets at the top of the cathode compartment, or is piped to any point where it is to be used. The caustic-soda solution flows out through a pipe *m* at the front of the cell.

29. Efficiency of Electrolytic Cells.—The efficiency of any electrolytic cell is stated in terms of current, voltage, and energy efficiency. **Current efficiency** is the ratio of the amount of caustic soda or chlorine produced by one ampere in 1 hour, to the theoretical amount of caustic soda or chlorine produced by 1 ampere for 1 hour. **Voltage efficiency** is the ratio of the theoretical voltage (about 2.3 volts in the case of sodium chloride) to the voltage actually used. **Energy**

efficiency is the ratio of the theoretical energy or power required for the electrolysis, to the energy actually used, the energy actually used being the product of actual volts multiplied by actual amperes used by a cell. The energy efficiency is the important characteristic to consider. It is the electrical energy or kilowatt-hours per pound of product made, based on the operating results during a period of a year or longer, that determines the economy of a cell. A well-designed cell, such as that described, should consume about 1.2 kilowatt-hours per pound of caustic soda produced. The chlorine produced in such a cell is converted into bleach with lime and the caustic soda is concentrated and used in the mill, or sold if there is sufficient in excess. The electric generator or dynamo generally used is one which will deliver a continuous current of large volume under moderate voltage, say about 1,250 amperes at 120 volts. The cells are usually arranged in multiple, and the current is conducted to them through large copper conductors.

30. Treatment of Bleached Stock.—The bleached and washed stock, in case the washing is not done in the beaters, is transferred to the agitators or mixers, to the wet machine, or directly to the beating engine. The object of the agitators or mixers, which are large, cylindrical tanks provided with agitators in the center and capable of holding several bleachers of stock, is to furnish uniform stock for the beaters, in case the pulp is worked right up into paper. The stock is continually passing in and out of the mixers. The wet machine is used in case it is desired to transfer, or ship, the pulp in laps or to weigh the amount used in the beaters. In modern practice this plan is usually followed in the treatment of the sulphite fiber, after which it contains about 65 per cent. of moisture. The pulp is transferred to the beater in laps, but is opened out before it is placed in the beater. The stock is pumped directly from the drainers to the beaters, where it is finally mixed for the paper machines.

31. Chlorine Bleach.—Recently, since highly purified chlorine gas has become an article of commerce, being sold

in liquefied form in steel cylinders, considerable work has been done on the bleaching of pulp (and also textiles and other materials) with liquid chlorine instead of bleaching powder. Very pure liquid chlorine is now being placed on the market by several firms at a reasonable price. It is shipped in steel cylinders containing from 100 to 150 pounds and can also be shipped in tank cars containing 30,000 pounds. The cylinders measure 53 inches by $8\frac{1}{2}$ to $10\frac{1}{2}$ inches. The following is an analysis of the chlorine sold by one of the leading manufacturers of this product: Chlorine, 99.80 per cent. to 99.99 per cent.; carbon dioxide, .01 per cent. to .20 per cent.; air and oxygen, .00 per cent. to .10 per cent.

The use of liquid chlorine obviates all the labor, trouble, and uncertainty of making up bleaching liquors from bleaching powder and water. Highly efficient, special proportioning valves have been devised, that will automatically admit enough chlorine to the water to bring the solution up to any required percentage of available chlorine. The convenience and simplicity of this method is rapidly becoming more and more appreciated and undoubtedly will be used to an increasing extent in the future.

BEATING

32. Beating Process.—In the beating process, the material is disintegrated in order to obtain a close, even sheet of paper. The amount of beating required varies according to the nature of the stock and the class and grade of paper to be manufactured. The beating process is one of the most important steps in the operation of paper making. No amount of skill of the paper maker will remedy a mistake due to carelessness or lack of skill on the part of the beater man. The manufacture of paper, as distinct from the manufacture of pulp, starts in the beater room. In studying the preceding sections, dealing with the various processes for making pulp, it should always be kept in mind that pulp is not paper—it is merely one of the raw materials of paper, of which there are a number of others of lesser importance, clay, size, colors, etc. There are many large paper mills which do not manufacture

any pulp at all, but buy all their raw material from other plants which stop with the manufacture of pulp and do not proceed to make it into paper.

33. Beaters.—The beaters, or beating engines, are large, oval, tank-like machines constructed of 3- or 4-inch cypress or other suitable planks. A usual size is about 25 feet long by 11 feet wide. Such a beater will hold about 1,500 pounds of completed stock. The usual height of the walls of the beater is about $3\frac{1}{2}$ feet. Some beaters are constructed of iron, having cast-iron or steel-plate sides and ends, and a bottom of

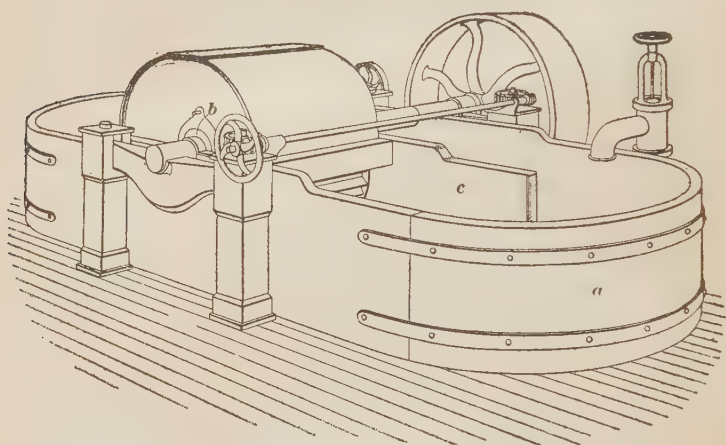


FIG. 3

wood, cast iron, or concrete. Concrete beaters are also used. In Fig. 3 is shown a typical beater. Extending through the middle of the tank *a*, parallel with the sides, but stopping short of the ends by about 3 feet, is a sturdy fence-like partition *c* called the midfeather. The midfeather may be of either wood or iron. On one side of the tank, filling the space between the midfeather and the wall, is a cylindrical beater roll *b*. This roll is so proportioned that its diameter is about equal to its length, the exact dimensions varying with the size of the beater. This roll is equipped with 78 steel bars or knives, each about 8 inches wide and $\frac{1}{4}$ to $\frac{1}{2}$ inch thick. At the bottom of the tank, directly under this roll is a bed-

plate, extending the full width of the roll and shaped so that its upper surface is parallel with the surface of the roll. The bedplate is usually about 16 inches wide and 5 inches high, and it contains 42 knives. These knives are not set exactly parallel with the knives of the roll. They are usually arranged at an angle or in a V-shaped arrangement.

34. A beater like that just described is frequently spoken of as a *hollander*, or *Holland type beater*. Other special types of beater, but intended for the same purpose, have been designed. A few of the more important of these will be described later, but the foregoing covers the usual type of beater in paper mills throughout the world. When the beater roll revolves, each of its 78 roll bars or knives comes in contact

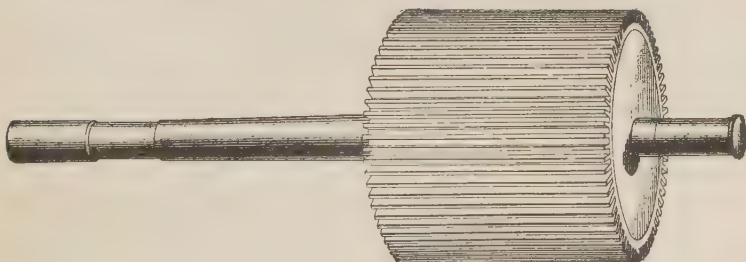


FIG. 4

with each of the 42 knives of the bedplate, so that the roll will give 3,276 cuts for each revolution. With the roll running at a speed of 100 revolutions per minute, it is apparent that there will be delivered 327,600 cuts per minute to any material forced between the roll and the bedplate. The beater roll, shown in Fig. 4, weighs several tons and may be raised from, or lowered upon, the bedplate by *lighter bars*, which carry the roll in strong journals. Such movement is rendered very accurate by having a hand wheel geared to it in a rather large ratio. Thus the roll may be changed in its relation to the bedplate a very small fraction of an inch. These fine adjustments are frequently necessary.

35. The beater helps to feed the various ingredients of the paper as determined by the formula. When it has been

found by experience that a certain percentage of sulphite pulp, a certain percentage of kraft (sulphate) pulp or of ground wood, so much coloring matter, so much *fixing* solution, size, filler, etc., is required to produce a certain definite grade of paper, these percentages are rigidly adhered to every time a run of this paper is made.

Naturally, such measurements and chemical treatment cannot be carried out by guesswork. The percentage of the various ingredients are reduced to terms of weight (the various pulps, sulphite, sulphate, and ground wood being fixed on a *5-per-cent. air-dry basis*, and the *furnish*, this being the phrase by which the exact formula of a given grade of paper is known, is built up accurately on exact scales. When a large mass of pulp is placed in the vat (which is capable of holding 1,500 pounds of completed paper stock) the revolving of the roll will draw the material between itself and the bedplate and cause a general circulation of the material, around and around the tank, since the midfeather gives a perfectly oval path for the *stuff* to travel in.

36. A short distance in front of the roll, a small box or trough extending from the side of the beater to the midfeather, is set in the floor of the beater. This trough is covered with a screen plate. It is known as a *sand trap* and it serves to eliminate small particles of grit and dirt which, on account of their weight, stick to the bottom of the stock as it circulates around the beater. This is used only for fine papers.

As the beater roll turns up the material rather sharply behind it, a cover or decking is necessary at that part, and a little beyond, to hold the mass down to its proper level and prevent its being thrown out of the beater. Immediately behind the beater roll is a device known as the *backfall*. This is a hump or elevation constructed of wood covered with steel plate, the side of which nearest to the beater roll conforms in shape to the roll. The pulp is propelled upwards between the beater roll and the *backfall* and strikes the cover of the beater which gradually smooths out the flow so that the pulp again becomes level in its travel around the beater before coming

under the beater roll the next time. The tendency to mount up and overflow behind the beater roll is much more marked in the case of the heavy viscous stocks—such as those containing a high percentage of kraft pulp.

37. Directly above the backfall and behind the roll is a *doctor*, consisting of a heavy board with a cast-iron edge. It is arranged so as just to clear the surface of the roll and prevent any stock from being carried around with the roll, deflecting it back over the top of the backfall.

In beating certain classes of stock such as waste paper, jute, etc., strings, rope, wire, etc., are frequently present. The *string catcher* is a device consisting of a series of bars or fingers secured to a steel shaft which extends over the path of the stock in the beater. The shaft and fingers form a sort of fork or rake which will catch any pieces of string or rope without seriously impeding the flow of the stock. A hand wheel is provided by means of which the device can be raised or lowered, as required. This device not only prevents string and rope from being mixed with the stock but also prevents such material from winding around the beater shaft where it would cause a great deal of trouble.

38. Various arrangements are used for emptying the stock from the beater. One of the commonest is an iron disk resting on a circular seat. In the center is a depression spanned by a cross-bar by means of which the disk is lifted out of its seat, allowing the stock to flow out of the beater. This device has the objection that the opening is relatively small, thus increasing the time necessary to empty the beater. Moreover, if any pressure is created in the stock chest, for instance, when one or more other beaters are being emptied, the disk may be forced up, allowing the contents of the beater to be prematurely dumped. This is guarded against sometimes by a lock type of valve. However, this is no more rapid in operation than the ordinary type and involves fumbling with a locking device at the bottom of a beater full of stock.

Much better are special quick-emptying valves, which are operated by a lever outside the beater. The disk is carried

on a riser operated by a series of levers. A spray of water that assists in the rapid removal of the stock and also keeps the valve seat clean and free is also provided with this type of valve.

39. A still further improvement is a patented stock-emptying valve of oblong shape about 6 inches wide located in the bottom of the engine just in front of the roll, extending right across from the midfeather to the side of the beater, its top being flush with the bottom of the inside of the engine. The valve cover lifts up along the side farthest from the roll, and when raised to a vertical position becomes a dam, aiding in forcing the stock out of the beater. It is operated by a shaft extending through the side of the beater and having a lever at its outer end. A shower of water is provided to help the stock out of the beater and to keep the valve seat clean. With an adequate discharge pipe (not less than 18 inches in diameter), the stock can be removed from the beater almost instantly without using any rakes and with a minimum of labor. With the ordinary disk valves the use of rakes is generally imperative.

40. **Function of Beater.**—Referring to the roll and bedplate of the beater as containing *knives* may possibly cause one to think that the main purpose of this machine is to cut the fibers to a given length. While the machine admittedly is used for this purpose, this is not its only function, or its most important one. No less vital than the cutting of the fibers to a certain length, is the separating of the bundles of fibers (which will exist to a certain extent in even the best grades of pulp) and to brush or stroke the fibers into greater flexibility. The tiny fibers are stroked out by the blunt knives of the beater, in somewhat the same manner that a hair brush strokes out human hair, and the fibers are caused to curl at the ends.

Upon the ability of these fibers to curl and connect with each other, when allowed to *bond* (by removal of the surrounding liquid) depends the strength and toughness of the resulting paper. If these fibers are not drawn out to the

their length, but they will be considerably weakened, and the sheet will have a raw, soft feeling. Such stuff is generally termed *fast or free*.

In brief, the beater is not an automatic machine. It is an instrument—and one that requires intelligent and experienced control. An inexperienced or careless operator can easily ruin an entire charge of material—especially during the first hour and a half.

43. The *rawness* just alluded to as caused by putting the roll too sharply down on the plate at first, is often quite noticeable in papers made from kraft pulp. In the thin papers of this class, the idea that length of fiber necessarily means strength is so often over stressed that, when the pulp reaches the paper machine, a considerable portion of it refuses to drain properly.

In preparing stuff for thick papers, the roll can be put down much sooner after the beater has been furnished. This is so that the stuff may be fine and free, parting with water readily on the paper machine, and giving a close, easily felted sheet.

44. Blunt plates and rolls are used for stock intended for thin, strong papers, which must be kept in the beater for at least 6 hours, during which time sharp knives would cut it up altogether too much (making it too free) and preventing it from felting properly on the machine. For preparing such stock the Jordan and other engines, subsequently to be described, are very useful; the stock being beaten a shorter time and finished in the Jordan. In the thick, heavy papers, sharper plates and rolls may be used, and the stock is not held nearly so long in the beater. For instance, stock for blotters is frequently kept in the beater only an hour and a half.

From these considerations it will be understood why the beating time varies so widely in preparing materials for different grades of paper, ranging from merely mixing the stock and color and then dumping in 30 minutes, to combing out and beating for 8 hours or more. It is always preferable to use separate beating engines for the extremes of adjust-

ment just illustrated. Or, in other words, the beater that is used for greasy, slow stuff should not be used for short ground-wood fibers. With even the most careful manipulation and adjustment, there is a certain range over which a beating engine operates most efficiently.

45. In the manufacture of some grades of newsprint, with a large percentage of ground wood, a treatment in agitators sometimes precedes the treatment in the beater. These agitators are large tanks provided with mechanically driven stirrers to keep the stock in circulation. The pulp is treated with size and alum, and held in the agitator sufficiently long for the size and alum to penetrate the fibers. This is called *soft stock* as opposed to sulphite, which is *medium stock*, and kraft, which would be a good example of *hard stock*. Numerous forms of testing instruments have been devised for determining the control of the beater operation, but the human element still governs it to a high degree. The appearance of the stock in the beater and the feel, when a handful of it is picked up, are the chief points on which the experienced operator relies.

Even the observing novice will notice that when the stock is first admitted to the beaters, it is cold, bulky, and fills the beater to the brim. This appearance will be kept up for some time, the stock breaking at various intervals, just before it passes under the beater roll. As the operation proceeds, however, there is a slight rise in temperature, the stock tends to sink more to the bottom, and at intervals it will shine on its surface.

After the stock has been worked in the beater it has a characteristic feel, quite different from that of unworked stock. The hand will pass through it freely and it will be slippery and greasy so that it is practically impossible to retain any large amount in the hand when squeezed. An experienced paper maker, by taking up a handful of stock in a beater, can generally tell how long the stock has been beaten, and how long it will still have to go, from the feel alone, even if he has no other source of information.

46. Broke Beater.—The *broke beater*, as its name suggests, is used in working up *broke*, which is partly formed paper obtained when starting the paper machine, paper damaged in passing over the drying cylinders, and imperfect or rejected paper. This engine is the same as the ordinary beating engine, except that it has a steam line by means of which the stock is highly heated. In some cases, a little caustic soda is added to the stock to assist in breaking it up again.

47. Jordan Engine.—Figs. 5 and 6 will serve to explain the Jordan engine. This machine consists essentially

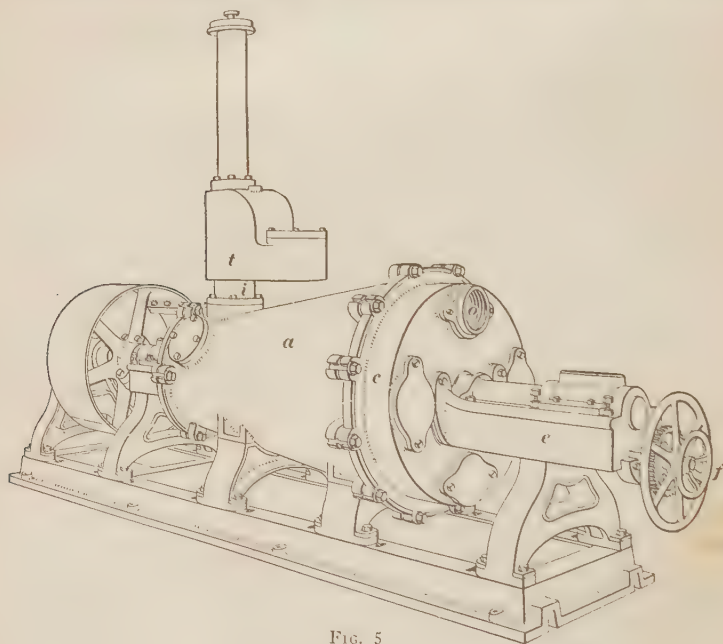


FIG. 5

of a conical cast-iron shell *a*, the inside of which is fitted with long, narrow steel bars, and rotating inside this conical shell is a conical casting *b*, called the *plug* or *runner*, the outside surface of which is fitted with long, narrow steel bars, or *knives*, each resembling, more or less, the runner of a skate,

although only about $\frac{1}{4}$ inch high and about the same width. The runner is journaled to rotate about its long axis, and, like the beater roll, can be adjusted to clear the inside plates of the shell by a very minute distance. This adjustment is performed by means of a hand wheel *f* and screw. The runner makes from 300 to 350 revolutions per minute. The plate *c* is bolted on tightly and the packing gland *d* is adjusted over the shaft. The arrangement as shown at *e* is attached as shown in Fig. 5 and supports the screw and hand wheel *f*. At *i* is seen the entrance for the stock, and at *c* the outlet.

The bars, or knives, both on the shell and the runner, are accurately ground, so that when the runner is properly adjusted, each knife cuts its entire length.

But this contact of the runner with the inside knives is not a direct or right-angle cut. The bars are so arranged as to

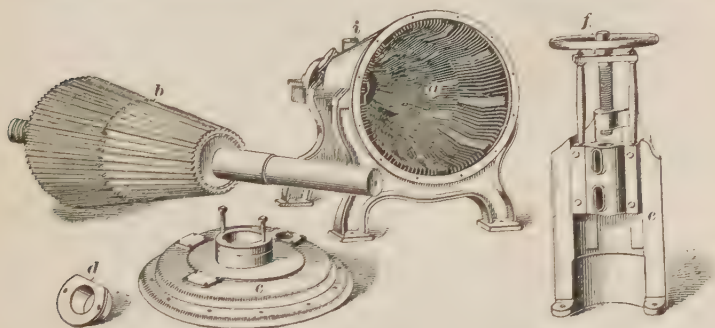


FIG. 6

deliver a shearing cut to the material, in somewhat the same fashion that the blades of a lawn mower are obliques against the bedplate. These engines are massive affairs, weighing several tons, and requiring a driving power of from 75 to 250 horsepower, depending on the grade of paper being made. Kraft rag and jute stock require considerably more power than any other kind ordinarily met with, on account of their long fiber and heavy consistency.

48. The Jordan engine gives the paper stuff the last refining touch before it goes to the paper machine. Each little bundle of fibers, which would otherwise clog and mar

the final result, is separated and distributed throughout the material in such a manner as to make the whole stock consistent and homogeneous.

The material is forced into the small end of the cone, and out through the other end, having been compelled to pass through the very small space between the plug and the shell, and between the whirling knives. From the large end of the Jordan it passes down to a second stuff chest, quite similar to the one that receives the stuff from the heaters, and is maintained in suspended state until the paper machine is ready for it.

At the inlet to the Jordan a sand trap *t*, Fig. 5. should be provided to keep sand and grit and also foreign matter, such as nails, screws, pieces of iron, etc., out of the Jordan. On

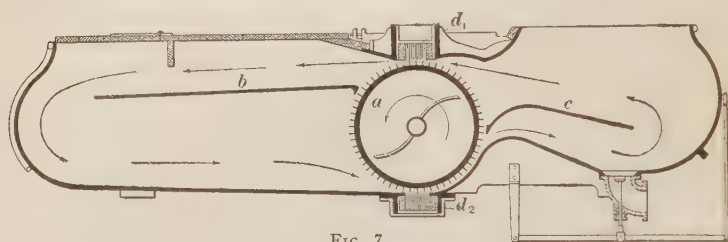


FIG. 7

account of the nature of the Jordan, such material would soon play havoc with the machine. It is important that these traps should be kept clean, otherwise they are of no use. Sometimes a powerful magnet is built into the sand trap to retain pieces of iron. This is a very useful device.

49. Marshall Engine.—The Marshall engine is another refining engine, more used in England than in America, but very useful for making certain classes of paper. It is especially good for preparing long, strong stock for thin, tough papers. It is quite similar to the Jordan, except that after the stuff has passed between the cone and the shell, it is caused to pass between a revolving and a stationary disk, both being provided with knives or bars. The revolving disk is attached to the end of the plug and the stationary disk is fastened to the inside of the shell head. The disk has a brushing action

of the fibers much like the action of the roll in the beater, and the stock emerges from the machine free from chips and bundles of fiber and of uniform consistency.

50. Miller Duplex Beater.—This beater is designed on the principle of effecting two beating operations for every circulation of the stock through the tube. Fig. 7 shows a section of the Miller beater, the submerged roll *a* and the front and rear midfeathers *b* and *c* dividing the tub into upper and lower sections, through which the stock is circulated by contact with the roll in both sections, which greatly increases the rapidity of the circulation and makes it impossible for the stock to settle or lodge in any portion of the tub. The

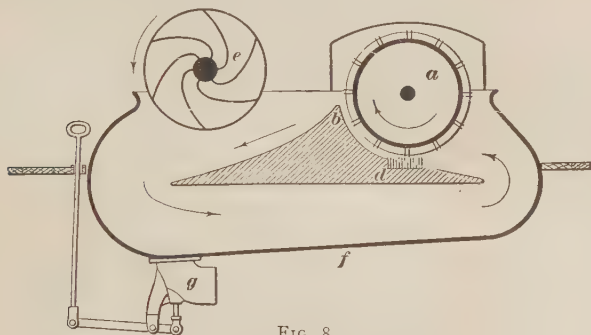


FIG. 8

location of the bedplate d_1 and d_2 both below and above the roll, doubles the beating capacity of the engine. The roll and the top bedplate are carried in double lighters. The bedplate lighters are connected to and controlled by the roll lighters in such a manner that the top plate is raised and lowered twice as fast as the roll, thus preserving an equal distance between the roll and both plates. This beater can be equipped with one or more cylinder washers just as in the case of the usual type of beater.

51. Umpherston Beater.—In the Umpherston type of beater, shown in Fig. 8, the stock is caused to pass below the floor *f* and backfall *b* on its return path to the front of the roll *a*. The bedplate is shown at *d* and a washer at *e*. At *g*

is the discharge valve. This machine is very economical of floor space and it is also supposed to give very perfect circulation with somewhat less expenditure of power than the usual type of beater. However, although many of these machines are in successful use, as is also the case with numerous other specially designed beaters, the ordinary type of beater will be found in the majority of mills. The Umpherston beater can be equipped with cylinder washers in the same manner as any other beater. The illustration shows an engine with one washer.

52. Claflin Continuous Beater.—The Claflin engine affords a means of making the beating operation continuous, which is an advantage from many points of view. At the same time the Claflin accomplishes everything that the Jordan engine can do. Consequently a suitable installation of Claflin engines may take the place of a combination of beaters and Jordans. This substitutes one type of machine for two, cuts down the power consumption (since the Claflin requires less power than the Jordan) and at the same time affords a continuous process.

Claflins are most usually installed for preparing stock requiring severe beating treatment, for instance, kraft stock intended for the manufacture of high-grade bag and wrapping paper.

The cone of the Claflin engine is much more obtuse than that of the Jordan engine, a fact which accounts for the lower power consumption. The arrangement of the knives is also peculiar to this engine and has been carefully planned so as to yield the same thorough brushing out of the fibers in ordinary beating engines when operated in the proper manner.

SIZING, LOADING, AND COLORING

SIZING

53. Varieties of Sizing.—It is necessary that writing paper, book paper, etc. shall not readily absorb ink or water, so that when used for writing or printing the ink will not spread, but will leave good, plain characters. This property is imparted to the paper by the use of what are known as sizing agents, which are assisted to some extent by the fillers or loading agents, which will be considered later.

Several different materials are used for sizing. Rosin size, however, is at present by far the most usual.

Animal sizing: Animal size was introduced in the days when paper was still made by hand. It is still used in England and in certain mills in America which produce fine writing and drawing papers. This size is really a solution of gelatine, prepared by soaking hides in water. It was applied to the paper after the sheets were made, by dipping the sheets in a vat of the size.

In America today so-called animal sizing is done with solutions of commercial glue and gelatine, and the paper is led from the machine through a trough or vat containing the size. These machines are usually called *size presses* and are placed in the dryer part of the paper machine, the dryers being separated into two nests.

The drying of animal-sized paper is an operation requiring great care. It must be carried out slowly and at a low temperature. Frequently such paper is *loft dried*, that is, the sheets are suspended on poles in a warm, dry loft. This treatment brings out very fine qualities in the paper. When loft drying is not resorted to, sometimes special forms of mechanical dryers are used in which the paper is festooned in a blast of warm air, as in a coating mill.

Engine sizing: Engine sizing is the term applied to the addition of size to the beater where the stock is being prepared for the paper machine. It is the usual method of sizing. The size most usually added is rosin size.

Rosin sizing: Rosin is a resin obtained in the manufacture of turpentine spirits from crude turpentine, which is a natural product obtained from pine trees. There are numerous grades of rosin, these grades being determined by the color. The grades are distinguished by letters of the alphabet. Rosin is graded B, C, D, E, F, G, H, I, K, L, M, N, W-G (window glass), and W-W (water-white). B is the darkest and W-W the lightest grade. Ordinarily the first three grades, B, C, and D, are not separated. Grades E, F, and G are the ones usually employed for making size in the paper industry. The other grades are used in other lines of manufacture. Rosin is sold in rather peculiar units of 280 pounds. This is derived from the English gross ton. However, the 280-pound barrel includes the weight of the container.

Provided that the darker color is not harmful, it is better to use E or even D rosin than the lighter F and G, as the sizing value is higher. However, D and G are the limits and should not be exceeded in either direction.

Rosin is a weak acid and will combine with an alkali to form a chemical compound, known as a *resinate*. In making size the rosin is made to combine with sodium carbonate or soda ash. Usually 58 per cent. soda ash is used. Rosin and soda ash do not react until heated. When mixed and heated they combine, carbon dioxide gas being given off, which gives rise to foaming.

54. There are many ways in which rosin size may be prepared, but they all accomplish the same purpose, that is, getting the rosin in such condition that it can be made into a solution from which it is again precipitated as already suggested. There has been a great deal of discussion as to what the true sizing agent is, some of the authorities contending that the free rosin is the only sizing agent, others that the resinates of sodium and aluminum are the true sizing agents, and still others that it is due to both. Practical experience inclines to favor the view that the sizing is as much due to resinates of aluminum as it is to the free rosin, as sizes in which the free rosin varied from 3 per cent. to 35 per cent.

have been successfully used. When using a size containing 3 per cent. of free rosin, a larger amount of alum was required to precipitate the size, but no more rosin was required to furnish a hard-sized paper than when 35 per cent. of free rosin was used. The chief advantages of using a size containing a large amount of free rosin are the saving of considerable soda in making up the rosin soap and the saving of an enormous amount of alum or other precipitant used. It is quite probable that some classes of paper are sized best by means of free rosin, while others are sized best by resinate of aluminum.

The usual method of making the size is to dissolve the soda ash in water in a kettle heated by a steam coil or a jacket. Sometimes the kettle is heated by direct steam, but this is not good as it is necessary to make allowance for the dilution from the steam in weighing out the materials for the size. When the soda liquor is ready, the finely powdered rosin is stirred in and the whole boiled for some time, after which it is diluted with water.

55. The proportion of soda and rosin used varies. The maximum amount of free rosin can be obtained by using 9 pounds of soda ash and 100 pounds of rosin. Such size is rarely used. From 20 to 40 pounds of soda ash per 100 pounds of rosin is quite usual, although some prefer 15 to 18 pounds of soda to 100 pounds of rosin. The soda and the rosin never combine completely. That is, there is always free rosin and free soda in the size, even if just the right quantity of soda for the rosin is added. The amount of free soda and rosin decreases, the longer the size is boiled. No rule can be given for the percentage of free or of combined rosin that a size should contain. It depends on the condition under which a size is to be used, the nature of the stock, the water, etc. A size that will work well in one mill may be useless for another.

After cooking until the lumps of rosin are dissolved and the batch is of a clear dark color, when the steam is turned off and the foaming subsided, a rough test is made as follows: Take a $\frac{1}{2}$ pint of hot size, add to it a quart of hot water and

stir until well mixed; now add sufficient cold water to fill a pail, and stir again. The resulting liquid should have a white or yellow color and dissolve to a thin milk free from lumps, grains, or sticky pieces of rosin. If it does not readily mix with water and dissolve to a milk, but forms grains like corn meal, it must be again cooked, but the cooking must be stopped when the test shows it to be done, as further cooking injures the size.

After it has stood for a day or two a black liquor separates which is brine and soda ash. This should be removed, as it causes foaming on the screens, etc., and the running off of the black matter is of a great advantage. By longer standing and occasional poking with a stick, more liquor can be caused to separate from it, and more should be worked off.

56. Ready-Made Versus Mill-Made Size.—Ready-made size is undoubtedly more convenient than making size at the mill, but it is much more expensive, and in spite of all the mystery surrounding the subject, any practical paper maker should be able to learn to make size suitable for his particular class of paper after a little experimenting.

Various Kinds of Rosin Size.—The following are descriptions of some of the kinds of size the paper maker will find, or will be given recipes for making, in many mills.

Highest free-rosin size: An example of highest free-rosin size is one of the ready-made sizes that contains so much rosin that, in order to get it into solution properly, 50 gallons of water must be used to dissolve 1 gallon of the size. One-third of the 50 gallons is boiling hot and the hot size is sprayed into this water by means of a steam injector. The other two-thirds of the water is run in cold. If care is not exercised the result is a sticky, unmanageable mass. There is no doubt that such size gives good results and is economical of alum, but it is troublesome and in the long run the economy is doubtful.

Second-highest free-rosin size: Second-highest free-rosin size is another group of sizes on the market containing less free rosin and capable of being dissolved without the need of

special appliances, but still requiring great volumes of water for solution and being very troublesome to handle.

Most popular size: The most popular size generally is that which contains a large amount of free rosin, but which any quantity of water will dissolve.

Old-fashioned size: Many old-fashioned size makers still adhere to the practice of using excessive quantities of soda ash and cooking the size so thoroughly that all the rosin is converted into soap and no free rosin remains. It is tested by dissolving in water, and if the size gives no yellow-milk color they cook it again. This is very wasteful of soda ash and alum. Old-fashioned size makers call modern high free-rosin size, raw size.

57. Alum.—Alum, in the usage of the paper maker, does not mean the alum of the chemist, which is the crystallized double sulphate of aluminum and potassium. Paper makers use the term alum to denote aluminum sulphate, $Al_2(SO_4)_3 \cdot 18H_2O$. The true alum was once used in paper making, but has been replaced by aluminum sulphate which is cheaper and stronger in alumina. The aluminum sulphate or paper makers' alum of commerce is not a definite chemical compound, different makers driving off different amounts of the combined water before the product is placed on the market. The best commercial product on the market today contains 22 per cent. alumina, Al_2O_3 , which is equivalent to 73 per cent. of aluminum sulphate, $Al_2(SO_4)_3$.

Commercial alum is generally prepared from bauxite, a naturally occurring hydrous oxide of aluminum. Pulverized bauxite is agitated with 50° Baumé, sulphuric acid in lead-lined tanks. The mixture becomes very hot during the reaction, after which it is diluted with water, allowed to become cool and to stand long enough to deposit silica and other impurities. The clear liquor is then decanted off, concentrated with heat, and when thick enough, run out onto marble slabs, where it crystallizes in a mass which is broken up, packed, and sold.

58. As bauxite always contains some iron, the commercial alum is rarely free from iron. The iron is frequently reduced to

the ferrous condition with zinc before the alum is crystallized, but this procedure is useless from the paper maker's point of view as the iron soon oxidizes again and colors the paper in which it is found. A good alum should contain very little insoluble matter, not over one-half of 1 per cent. More insoluble matter indicates that the alum has been made in a hurried and careless manner. Free sulphuric acid is objectionable in an alum. It decomposes the size, corrodes the wire of the paper machine, disintegrates the felts, and attacks every pipe and vessel with which it comes into contact. It is possible to obtain alum from the manufacturers quite free from free sulphuric acid and this should be insisted on.

59. Function of Alum in Paper Making.—It is frequently said that alum is added to size the paper. That is not correct. The true purpose of alum is to precipitate the dissolved rosin in the size on the fibers of the paper. This is sometimes called *setting* the size. It also *sets* or *fixes* certain colors, making them stronger and brighter, as certain reds and blues. However, it weakens certain other colors, such as yellows and greens. If it is necessary to make a heavily sized yellow paper, it is best to experiment until the exact amount of alum to set the size is found and then to use care not to add any more than that, as it will weaken the color. This also applies to many other colors. However, many paper makers have the idea that alum strengthens all colors. They say that it keeps the color from washing out of the paper, and when they are making any highly colored paper they are very liberal in their use of alum. This practice is unscientific, and in many cases is absolutely wrong.

Too much alum renders the paper brittle, causes it to lose moisture rapidly; and is a cause of rapid deterioration. Sometimes excessive amounts of alum are added to impart a stiffness and rattle to the paper, but this is bad practice, as it is responsible for the troubles previously described, and the desired effect can be secured in other ways; for example, by the use of silicate of soda, starch, etc.

60. Excess of alum is also hard on the wire and felts, especially the dryer felts. The rottenness of felts, frequently attributed to burning or scorching on the dryers, largely is due to the action of alum. The combined action of the alum and the heat of the dryer will soon destroy the felt.

In addition to setting the size, alum has a clarifying effect on the water. If water is turbid because of the presence of clay, etc., it cannot be filtered bright, as the slime will either pass through the filter or else it will completely stop up the pores of the filter so that water will not pass through at all. Alum causes the dirt in the water to coagulate so that it falls down in granules, leaving the water above clear and bright. In paper making the alum exerts this same property, and much clay and fine fiber that would otherwise be carried off with the water, is coagulated and held in the web and is less likely to be sucked out by the suction boxes or to drain through the *wire*. This is why alum aids in the retention of clay, a factor already alluded to in the discussion of the use of clay as a filler.

61. Silicate of Soda.—Silicate of soda is also known as *water glass*. It is generally sold in solution in barrels or drums. A 50-per-cent. solution is ordinarily used, but any required concentration can be obtained from the makers, or the solid silicate can be obtained. This chemical imparts a hardness and rattle to paper that makes it valuable for use in certain writing papers. It tends to set size very much like alum does. It should not be used in conjunction with alum, as the alum will yield a heavy precipitate with the silicate and thus cause trouble. Silicate of soda, on account of its adhesive and grease-proofing qualities, also is used in making certain boards and paper specialites such as corrugated container board.

62. Starch.—Various starches are used in the paper industry, such as corn starch, wheat starch, potato starch, etc. Starch is used for its hardening and stiffening action, and because it aids in the production of certain highly finished surfaces when the paper is calendered. Some paper makers

add the starch directly to the beaters and others mix it with the size. It seems to exert certain beneficial properties on the rosin of the size, enabling the particles of rosin to become better attached to the fibers of the stock. Corn starch is not the best to use, a mixture of starches being better, and 1 lb. starch should be boiled with 2 gallons of water and the mixture kept a little lower than the boiling point for 15 to 20 minutes, after which the starch is ready to be added to the beater. The retention of starch is about 50 per cent. The use of starch is confined mostly to certain high-class writing and book papers. A little is also used in cigarette paper.

LOADING

63. Materials Used.—In the manufacture of almost any kind of paper, except that of the very highest quality, it is customary to load the stock, that is, to add some comparatively cheap material, such as china clay, agalite, pearl hardening, etc., to the stock in the beaters, so as to give weight to the paper, to make it less transparent and to improve its surface. While, as has been stated, the appearance of a paper is greatly improved by loading, the strength of the paper is somewhat impaired, if too large an amount of filler is used.

The material most extensively used as a filler is china clay, or kaolin. This substance occurs naturally, being a hydrous silicate of aluminum, formed by the weathering and disintegration of certain kinds of rock. It occurs throughout the world, but until recently clay for the use of the paper industry has been procured chiefly from Great Britain. Lately, deposits of clay suitable for paper making have been opened up in the United States and Canada. The majority of clay deposits throughout the world are not suitable for the use of the paper industry because of the presence of impurities.

64. The composition of clay will always vary owing to the presence of impurities but a good average sample should run from 47 to 50 per cent. SiO_2 ; 34 to 40 per cent. Al_2O_3 ; 12 to

15 per cent. chemically combined water. The usual chemical impurities are iron, calcium, and the alkalies. Clay containing more than 1 per cent. iron should never be used, as iron will impart color to the paper. For making good white paper, clay should be perfectly white in color, very fine, and free from grit. In Table IV are given the analyses of four British clays, suitable for the use of paper makers:

Whether a clay is suitable for paper making, or not, cannot, however, be decided by chemical analysis, although all clays considered for use should always be analyzed to prove

TABLE IV
ANALYSES OF CLAYS

	I	II	III	IV
Moisture, loss at 100° C.30	10.15	7.09	9.10
Combined water, volatile at red heat ..	12.27	10.77	11.27	12.79
Silica, SiO_2	47.56	42.72	43.50	41.16
Alumina, Al_2O_3	38.12	33.44	35.48	35.84
Sesquioxide of iron, Fe_2O_308	1.04	trace	.67
Lime, CaO39	1.61	.17	.42
Magnesia, MgO00	.16	.41	.02
Alkalies	1.28	.11	2.08
	100.00	100.00	100.00	100.00
Specific gravity of dry substances	2.8625	2.5585	2.5451
Grit by flotation test (per cent.)65	6.8310

the absence of chemical impurities in excessive amounts. The physical properties of the clay are equally as important as the chemical properties. These physical properties are the result of the geological history of the clay. The chief point is the presence of what the chemist calls *colloids* in the clay. This means that the fine particles of clay will remain in suspension almost indefinitely when the clay is mixed with water. It is the presence of this colloid structure that gives the clay the greasy, slippery, tenacious consistency when mixed with a small quantity of water, which is characteristic of all good clays.

65. Clay is usually used as a filler in newsprint and the cheaper grades of writing and book paper. The better qualities of writing and book paper are usually loaded with special materials such as agalite, calcium sulphate, pearl hardening, barium sulphate, etc.

For use in the beaters, the clay is usually made into a thin cream with water, this generally being done in a small tank fitted with an agitator. Some paper makers mix the clay with rosin size before adding it to the beaters, it being believed that this procedure aids in the retention of the filler by the fibers.

Gypsum, or calcium sulphate, is used to some extent as a filler in high class papers, but on account of its slight solubility, it does not give so good a retention as do the other two fillers just mentioned. Gypsum is sold under various trade names, such as pearl hardening, crown filler, mineral white, etc. The retention of this filler in paper is only from 35 to 40 per cent.

Barium sulphate is sold under the name of *blanc fixe*, and an artificial compound of precipitated calcium sulphate and alumina is sold under the name of *satin or Salem white*. Both of these compounds are used as a paper coating.

Agalite is a filler chemically the same as ordinary talc, but of somewhat different physical structure, being prepared from a variety of talc that is more like asbestos. In fact, mineralogically, the talcs and the various sorts of asbestos are very closely related. On account of its fibrous structure, agalite is a very useful loading material. Just like talc, however, it makes the paper loaded with it very greasy. The terms *agalite* and *talc* are used very loosely and interchangeably by practical paper makers. Asbestine, French chalk, mineral pulp, etc., are all other names for the same thing.

COLORING

66. Methods of Coloring.—Coloring is effected by adding pigments or dyes to the stock in the beaters. There is a great variety of coloring matter that can be mixed with the stock to produce various shades in the finished paper. A yellow tint in the stock is neutralized by adding red or blue.

The blues generally used are ultramarine, smalt, Prussian blue, and various aniline blues. The reds are usually prepared from cochineal or aniline dyes, but as the latter are affected by the use of alum, cochineal red is to be preferred. Alizarine and red ocher (oxide of iron) are also used to a great extent in producing red tints.

Yellows are produced by the use of yellow ocher, lead chromate, and some of the coal-tar dyes, the principal ones being metanvl yellow and auramine. Browns are produced by the use of pigments of the iron oxides, or the Bismarck browns. Greens are usually produced by means of malachite green or Victoria green; blacks, by the use of lampblack, Frankfort black, or blue-black.

It is better to mix the coloring matters with water before adding them to the stock in the beater. The aniline colors should be dissolved in hot water and then diluted. Carmine should be dissolved in a little ammonia water and then diluted. Samples of the pulp treated are taken from time to time and matched against a sample of the paper that the paper maker is running, and the trained eye of the beater man can readily determine when the desired effect is produced.

67. In order to obtain the color desired, it is first necessary for the beater man in charge to try to match the color and then to calculate the amount of coloring matter required in the beater. This is best accomplished by mixing a definite amount of stock containing a known quantity of air-dry fiber with a convenient amount of water at about 80° F., and then adding the dye solution from a burette, using a 1-per-cent. solution. After this the stock is squeezed out and matched, the amount of coloring matter calculated, and the required amount added to the beater.

The water from the paper machine, or *back water*, or *white water*, as it is called, should be used over again, and the amount of water on the machine should be so regulated that very little of it will go to waste. In this way, there will be a great saving in the loading and coloring matter, which would otherwise go to waste. Before attempting to size or color the stock,

it should be perfectly cold; otherwise, good results cannot be obtained.

The coloring materials used in the paper industry may be divided into pigments, or mineral colors (however, a few pigments are non-mineral in nature), which are distinguished by being insoluble in water, and dyes, mostly artificial in origin and usually spoken of by the general title of *aniline dyes*.

68. Pigments color the stock by becoming enmeshed with the fibers in the beater. The size and alum helps the fibers to retain the pigments, which adhere in small particles to the surface of the fiber. Pigments do not penetrate the substance of the fiber as do dyes. There is probably **no** chemical action between the cellulose of the fiber and the pigment, whereas in the use of dyes, the combination seems to be more chemical than mechanical.

The percentage of retention of a pigment, and, therefore, the degree to which the paper is colored, depends on the manner of sizing, the amount of alum used, the specific gravity of the pigment and the nature of the stock. Slow stock gives a higher retention than free stock. The operation of the paper machine, whether or not suction-couch rolls are used, also affects the retention of pigment.

Pigments, if used in any quantity, have exactly the same action as clay. In fact, they may be considered as colored clays. Too much clay, as well as too much pigment will weaken the paper. Many pigments contain grit. All pigments to be used should be passed on by the laboratory to ascertain that they are free from grit, which will cause pinholes in the paper and also will injure the wire and felt and the calender rolls.

69. Dyes.—Aniline dyes, synthetic dyes, coal tar dyes or colors, etc., are all synonymous terms. They are all made from derivatives of coal tar by a series of complicated processes. The first such dye was invented by Sir William Perkin in 1856, since which date thousands of others have been invented. Dyes are divided into: (1) Basic dyes; (2) acid dyes; (3) direct dyes; (4) vat dyes.

Basic dyes are not very fast to light and are hard to use unless perfectly soft water is at hand. With hard water they give the paper a spotty or mottled appearance. These dyes require no alum to set them. Auramine is a typical example of these dyes.

Acid dyes must be set with alum. They are faster to light than basic dyes. Unlike basic dyes they will resist comparatively high temperatures without change. They also work better if the water is at all hard.

Direct dyes do not require any alum or other chemical to set them. They do not work well with hard water. They enter into direct chemical combination with the fiber and so are suitable for use with unsized paper, such as blotters. They are faster to light than either the basic or acid colors. These are the dyes that are known in the textile industry as *cotton dyes*.

Vat dyes are little used in the paper industry. They are practically pigments of synthetic origin. Only a few vat dyes have so far been made in America.

70. Irregular Coloring of Paper.—Fading of color often occurs where colored paper is exposed to light or is brought into contact with certain chemical substances. Care should be taken to select a color that will not fade when the paper is put to the use for which it is intended. The greatest difficulty along this line has been experienced in the making of colored paper for soap wrappers; it seems almost impossible to produce a paper the color of which will permanently resist the action of the caustic alkali in the soap.

Frequently, when pigments are used, irregularity of color of the two sides of the paper is noted. This is due to the fact that some of the coloring matter is drawn away by the suction boxes, and the side of the paper next to the wire is not colored so well as the upper side.

Unevenness of color is generally due to mixtures of various fibers that have different affinities for the coloring matter used. This is very marked when chemical wood pulp is mixed with mechanical wood pulp.

71. Coloring Paper With Dyes.—Dyes should be tested to ascertain whether a mixture is not being used. These do not give uniform and satisfactory results. A simple test for a mixture is to take a pinch of the dye on a knife or coin and blow it sharply onto a piece of filter or blotting paper dipped in water containing a little acid. If the dye is a mixture usually spots of individual colors can be seen where the tiny separate particles fall.

It is better to put the dyes into a liquid or a paste form before adding them to the beater. Dry dyes added to the beater are not so effective, as some of the dye is wasted and the effect is uneven. Basic colors and acid colors should never be used together. They tend to coagulate each other.

Basic colors should not be mixed or used together with direct colors. Acid colors can be used with direct colors, but not with basic colors. Excess of alum is bad for all colors. Just the right amount to use should be determined.

When a very full shade is desired, good results can sometimes be obtained by first dyeing the stock with an acid color and then submitting it to a second dyeing with a basic color. This will give a better color than could be obtained with an acid dye alone, and it will be faster to light than if a basic dye alone were used.

In coloring mixed stock, such as ground wood and sulphite, sometimes the ground wood will take the dye before any sulphite does, producing a mottled stock. This can be prevented by dyeing the ground wood stock and the sulphite stock in separate beaters and then mixing them.

Mottled papers, for instance, mottled blottings, are made by dyeing the stock strongly in two or more separate engines, then mixing just before the stock goes on the paper machine.

MANUFACTURE OF PAPER FROM PULP

72. Stock Chests.—In the basement of the beater room are a number of stock chests. These are for receiving the stock from the beaters preparatory to sending it to the Jordans or other refining engines, or to the paper machines. There are also other stock chests for receiving stock to be furnished to the beaters. Some of this stock is deckered stock that was received direct from the sulphite mill, and some of it is stock produced by disintegrating laps of kraft or other pulp with shredders and pulpers. Some lap stock is too hard and dry to feed to the beaters in this form, and has to be mechanically disintegrated before being furnished. This is usually done with machines called *shredders* and *pulpers*.

These stock chests, shown in Fig. 9, vary in size according to the room available, or the size of the plant, but an average storage chest will hold approximately 2.5 to 3 tons of air-dry stock. The chests *a* are provided with agitators *b* driven by a gear *c* and pulley *d*. The purpose of the agitators is to keep the stock of uniform consistency.

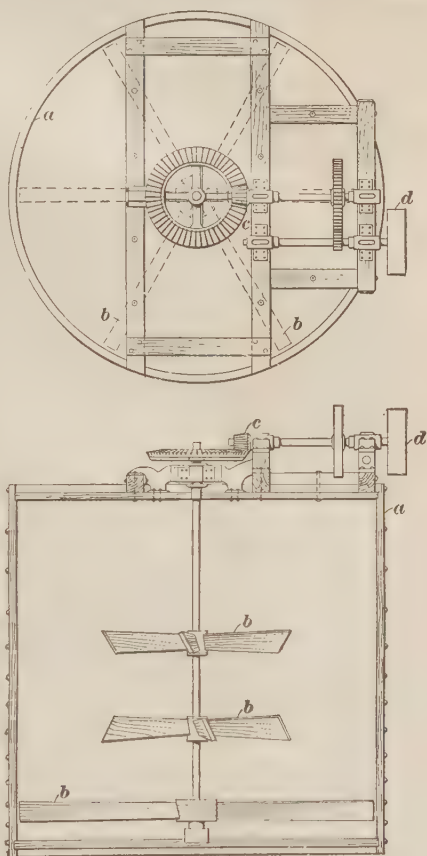


FIG. 9

The stock is pumped from the storage chests to the beaters by a centrifugal pump. It usually gravitates from the beaters to small chests located just above the Jordans. After treatment in the Jordans it is usually stored in other chests, equipped with agitators, called *stuff chests* or *machine chests* as they supply direct to the paper machines.

MAKING OF PAPER BY MACHINE

73. Paper Machine, or Fourdrinier.—The Fourdrinier machine, consists essentially of a device for allowing carefully screened pulp of constant consistency to flow onto a horizontal wire screen, made in the form of an endless belt and traveling constantly away from the point where the pulp flows on it. The water in the pulp drains through the wire, this drainage being assisted by suction boxes applied under the wire at certain points. At the end of the wire farthest from the point where the pulp flows on it, is a pair of rolls between which the film of fibers from the wire passes. At this point the film of fibers still contains much moisture, so it is passed through other felt-covered rolls, which press more water out of it. Next it passes through a long series of steam-heated iron cylinders, always supported by a layer of felt which travels with the paper, and these cylinders drive out all the remaining water except a small percentage always present even in paper commonly considered quite dry. Finally the paper passes through polished calender rolls to give it a *finish* and onto reels where it is wound up.

74. The foregoing description gives little idea of what a complex and intricate mechanism a Fourdrinier machine is, and of the necessity of having every part in perfect running order, and perfectly adjusted to every other part if satisfactory work is to be done.

The stock coming from either the Jordans or the beaters passes into a large cylindrical tank, called the *stuff chest*, which is made either of wood or iron, and is provided with an agitator that extends to the bottom of the tank and is kept moving at a

moderate speed. The stock is pumped from the stuff chest to the regulating box, which is a small box that is constantly kept filled by means of the inlet pipe at the bottom, the excess of stock being carried back to the stuff chest by an overflow pipe near the top. The discharge pipe leading to the screens is located near the bottom of the regulating box, so that there is a uniform pressure at all times. The amount of stock furnished can be regulated by means of a cock on this pipe.

75. The screens serve to remove any foreign matter that has escaped removal up to this point. After passing through the screens the stock should be free from lumps and dirt and ready for making into paper. Some paper makers prefer rotary screens to diaphragm screens because they believe that the advantages of the rotary type of equipment more than outweigh the somewhat increased complexity of the mechanism. The advantages claimed for rotary screens over diaphragm screens are as follows: With a fixed flat bed, screen plates cannot be maintained at a uniform or constant degree of cleanness, for flat plates accumulate dirt; they fill up and foul until it becomes necessary to wash up. Consequently, there is a period just before washing up when the stock is dirty. By building the screen plates in cylindrical form, as in the rotary or revolving screen, it is possible by a continuous shower to keep the plates clean all the time.

After leaving the screens, the stock usually flows into what is called the head-box, entering at the bottom, where it is mixed with more water, which is furnished by a supply pipe. This box serves to mix the stock well before it flows on the wire; this it does by overflowing the box. The head-box also serves to catch any heavy particles of matter, as they will remain at the bottom and can be washed out from time to time. Between the head-box and the slices *b*, Fig. 10, is a heavy rubber apron that fits over the wires and extends across the full width of the wire to within a few inches of the slices.

76. The apron is a shallow, flexible trough, through which the stuff flows onto the wire, thus bridging over the open space between the breast roll and the head-box. The slices, which

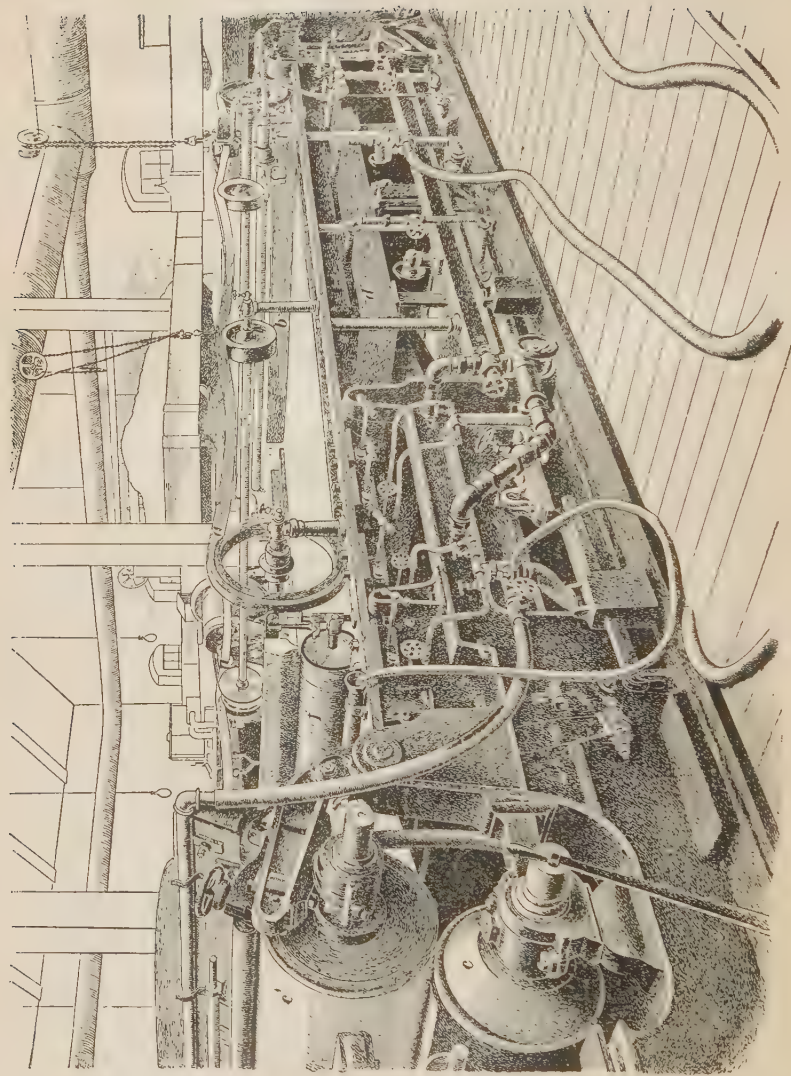


FIG. 10

are metal dams with perfectly level under edges set vertical to the surface of the wire, dam back the stock, filling the apron full of water containing pulp fibers. This is called the *pond*. The slices are raised 1 inch or less from the wire so as to allow all the water and fibers to flow onto the wire. The force with which the stock comes out of the pond and under the slices, is in direct proportion to the height of the water in the pond. This head is regulated by the supply of stock and also by the height of the slices from the wire. Without changing the stock supply, the slices can be lowered, reducing the area of the outlet, which results in backing up the stock in the pond to a greater height. This performance has the effect of giving the stuff more speed as it is delivered to the wire. Reversing this process gives the opposite results. These changes must be governed entirely by the kind of paper that is made. The stuff must be delivered to the wire at as near the speed of the wire as possible. The slices, in conjunction with the shaking of the wire, cause the fibers to be spread evenly and uniformly.

77. The slices when set very low catch the fibers and turn them on end, causing the sheet to have a broken, unfelted appearance. This is more noticeable when working long stuff, and it is the cause of the wavy streaked appearance of thick sheets. The thicker the sheet the more shake is required to felt it, and when making a thick paper, for which the stuff has been made long, the slices will have to be raised so that enough water may be worked in to assist in closing the fibers.

In starting a run the lower edge of the apron should be within about an inch of the first slice. This distance may often have to be varied later, depending on the particular kind of sheet being produced. When making a thin sheet from free stock, where there is difficulty, the moving of the apron clear down to the first slice will assist in carrying the water nearly to the suction boxes. Conversely, when making a thick sheet from slow stuff the apron should be pulled back, possibly as much as 2 inches, depending on conditions to be met.

The beaters are sometimes so full that it is not possible to add sufficient water as they are dumped into the stuff chest.

In such cases, the heavy stock goes to the bottom and is drawn into the pipe leading to the screens and thence to the head-box without being sufficiently diluted. On a heavy sheet of paper as high a difference as 5 pounds per ream will be caused by this variation in the thickness of the stock.

78. In order to overcome sudden changes in the consistency of the stock and to insure the production of a paper of uniform thickness and weight, the stock in a number of mills is passed from the stuff chest to a small box provided with a metal float. When the stock that enters this box becomes too heavy, the float rises and opens a valve through which fresh water is supplied to thin the stock. When the stock is properly diluted, the float drops and automatically closes the valve.

In order to insure a good supply of stock to the stuff pump, the pipes leading from the chests should not be less than 4 inches in diameter. This pump should be capable of pumping sufficient stock to insure a good overflow when the paper machine is working at its best. The thickness of the sheet is regulated by the slice.

79. The *wire*, as it is called, is really an endless wire cloth, closely woven and having from 60 to 70 meshes per linear inch. The length of the wire is from 35 to 40 feet, and the width is usually from 100 to 130 inches, though some are made as wide as 160 inches and as long as 75 feet. The wire passes around the lower couch roll *c*, Fig. 10, down under the save-all, and back to the breast roll. In its passage over the surface, the wire is supported by a large number of small brass rolls, called table rolls, and in returning to the breast roll, it passes over several small rolls. The frame has an attachment by which the wire is given a shaking motion from side to side, which serves to weave the fibers in their passage over the wire.

80. The pulp fibers must be thoroughly crisscrossed and interwoven on the wire while they are being formed into the film or web. This is the only place on the paper machine where the fibers can be interwoven and properly felted. Con-

sequently everything must at all times be in first-class condition. Assuming that there are only 15 to 20 feet of making-up surface (this length depending on the machine), on the wire, and with the machine running, let us say 500 feet per minute, it is evident that only a very small part of a minute is allowed for forming the paper. The speed at which the machine is to be driven, and the nature of the stock to be worked, must always be taken into consideration when specifying the length of the wire to be used. For the proper working of short, soft, greasy stuff, at the correct speed, a long wire is an advantage, thereby giving more time to allow the water to be taken out; but for fine, long stuff, not too soft, worked at a moderate speed, a short wire will be best suited. The speed must also determine the amount of pitch or inclination to be given to the wire.

81. Under the wire is situated the save-all, which is a shallow box *d*, Fig. 10, into which the waste water coming through the wire drops, and is used in place of fresh water for diluting the stock in the head-box referred to previously. Near the end of the wire and under it are situated the suction boxes *e*, four of which are shown in the illustration. These are long, narrow boxes, which extend across the whole width of the wire and are connected to a vacuum pump. The cover of the box, which is very smooth, is perforated, and the water is further removed from the stock by having the wire pass over it. There is a screw arrangement at the end of the box by means of which plugs may be moved forwards or backwards, according to the width of the paper, so that the boxes will not lose their suction. These plugs are always kept in as far as the deckle straps *a*, which are heavy, square rubber bands that rest on the wire and are carried along with it, thus regulating the width of the sheet. The dandy roll *f* is situated near the end of the wire, and is used for making the *water mark* in the paper. It consists of a skeleton roll covered with a wire cloth, upon which the desired design is worked with fine wire. If the paper is required to be alike on both sides, with no special design, the roll is covered only with wire cloth, the impression of which corresponds with the impression

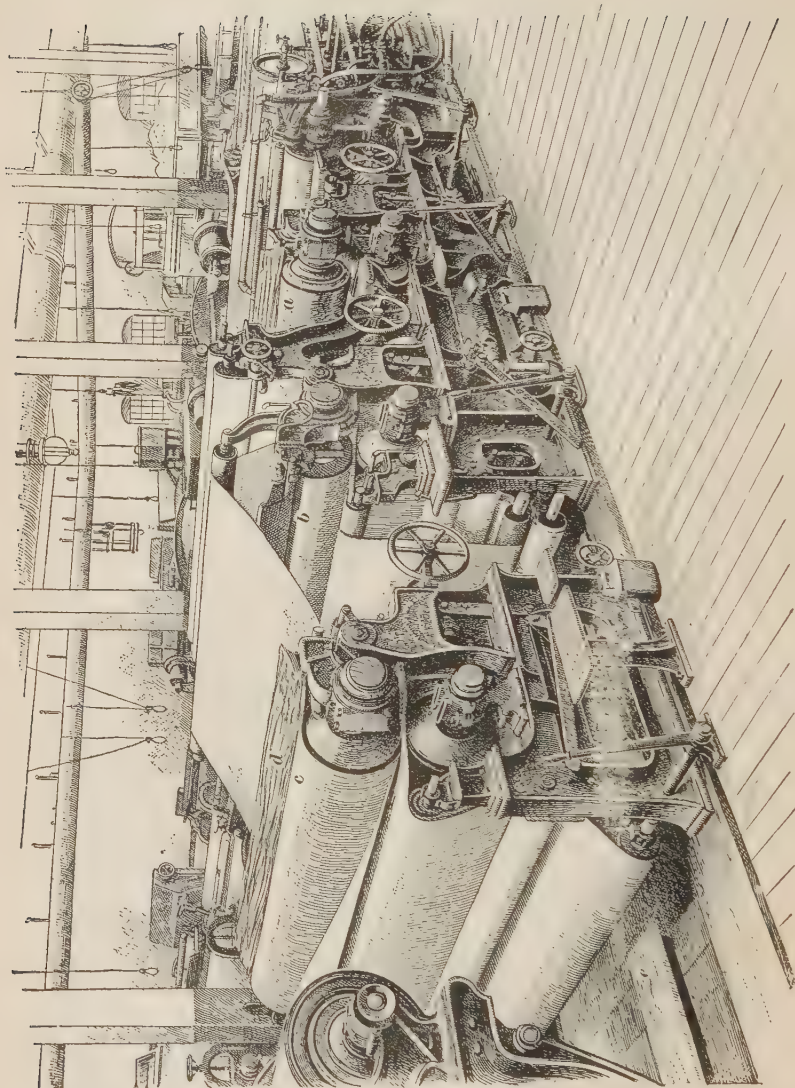


FIG. 11

on the wire cloth. Paper made in this way is known as *wove paper*. Laid paper is made by the dandy having a number of equidistant transverse wires upon its upper surface. The paper passes under the dandy roll and is carried by the wire cloth between the couch rolls *c, c'* which are brass or wooden cylinders with a jacket of felt. In some machines, the top roll is made of wood and the bottom roll is made of iron covered with a layer of heavy rubber. There is a system of levers and weights above the top roll by means of which the pressure can be regulated, thereby pressing the water out of the paper in its passage between the rolls. The paper is then carried, by means of felts and the assistance of the machine tender, through the press rolls proper. The number of press rolls varies on different machines, the machine shown in Fig. 11

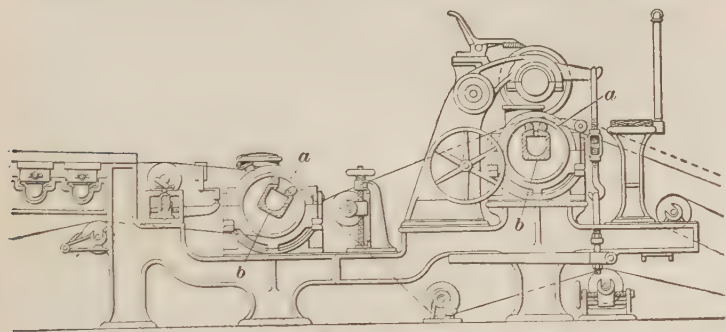


FIG. 12

having three sets, as shown at *a, b, and c*. The top roll of each set is provided with what is termed a doctor *d*, which keeps the roll clean by scraping off the pulp that sticks to it. The pressure on these rolls is also regulated by a system of levers and weights. After passing through the last press roll, the sheet is transferred by the machine tender across an open space to the driers, or drying cylinders; this space is wide enough for the machine tender to pass back and forth under the sheet.

82. Suction Rolls.—The suction rolls shown in Fig. 12, are specially designed rolls that take the place of the couch roll or one of the press rolls, and are intended to remove water

from the paper, by suction, in the same manner as it is removed by the suction boxes. These rolls also perform the usual functions of an ordinary couch or press roll. With the operation of wider and faster paper machines great interest has arisen in these rolls, and many modern paper machines are equipped with them.

The stationary suction chamber *b* is connected to a powerful rotary vacuum pump, usually located in the basement and driven from the constant speed line. The contact between the suction chamber and the inside surface of the revolving shell *a* is made with special hydraulic packing. A deckle arrangement is provided for adjusting the length of the suction area to accommodate any width of sheet made on the machine.

83. Wet Felts.—The process of making paper makers' felts, so far as the technical operations are concerned, is very much the same as that of making any woven woolen cloth, especially bed blankets. A paper makers' felt is in reality a blanket and not a felt in the strict sense of the term, the latter being a sheet of wool fiber so felted or pressed together as to present a smooth surface, but having no threads.

In making paper makers' felt, long, staple, strong wool is carded and spun into two kinds of yarns, the first being used for warp or threads lengthwise of the felt; the second is used for the filling or cross-threads. The warp gives the strength and necessary pulling qualities lengthwise. The filling makes the surface of the felt and from these filling threads the nap is raised.

84. The seam of the felt is made by hand. A long fringe of warp is left on each end when the piece of cloth is woven. The felt being placed on a table with the two ends together, the joiner ties a knot in the ends of the two exactly opposite threads and draws one out; the other following is drawn in and takes its place. This operation being repeated first on one side and then on the other makes a perfect hand-woven seam. The felting, fulling, or thickening of the cloth then takes place, and finally the raising of the nap. The coarseness of the

yarns, as well as of the wool, varies with the kind of paper to be made.

The wool fiber among all textiles is the only one against which a wet sheet of paper may be pressed to remove the water and from which this water may again be removed by pressure. It is the only fibrous material which will pick up a sheet of wet paper from some other carrying medium and deliver it again to any desired point without injuring the sheet and with no particles of the paper stock adhering to it.

85. In size, felts on the modern machine have gradually increased to lengths of 40, 50, and 60 feet. Here also, there is a great difference of opinion, but at the present time the majority of Fourdrinier machines are being built for first-press felts, varying in length from 40 to 60 feet—the second- and third-press felts a little shorter in some cases than the first.

The felt on the second press is sometimes of the same quality as that on the first, but usually heavier, while third-press felts are still heavier. The relative length of service of the first, second-, and third press felts on fast-running machines seems to depend on the amount of work in drying the paper that each is made to do. In other words, the heavier the pressure on the felt and the heavier the suction, the shorter is its life. Necessarily, with the great increases of speed met with in modern Fourdrinier machine operation, the service required of felts has become much more severe. At the present time a great strain must be put on the felts lengthwise—in other words, they must be run very tight—to keep them open, and admit of the high speed.

86. For tissue paper very fine felts with no nap are required, and owing to the peculiar conditions on tissue machines, the thinness of the paper and the necessary thinness of the felt, as well as the fact that felts become filled with stock very quickly and require constant hard beating, the felts probably cause more trouble on these machines than on any other.

The life of a felt depends very largely on the treatment it receives at the hands of the machine tenders. To meet the

conditions under which it is used, it must necessarily be a comparatively delicate woolen fabric, easily torn or injured, and yet it must do the work of an endless belt running constantly at a high rate of speed between heavy rolls, carrying a great weight of paper and water, and with constant liability to injury from unforeseen causes. There is no point in paper making where care and watchfulness count for more than in the treatment and use of felts. Felts in the stock room should be carefully watched and kept free from moths by the application of camphor, naphthaline, or tarred paper; they should also be kept free from all dirt and grease. The room should be dry and yet not hot.

87. Great care should be exercised in starting felts on the machine, since many felts are ruined by careless starting, whereas if a little precaution had been taken they could be made to give full and satisfactory service. The amount of service that can be obtained from a felt varies so greatly with different conditions that it is impossible to lay down any rule for this. Felts on two machines running side by side will sometimes differ 5 per cent. or more in the amount of paper made. It is possible for a felt to be injured in so many different ways that the felt itself should not be considered defective until every possible source of damage on or about the machine has been investigated and, in case the paper maker becomes convinced that the felt is defective in some way, it should never be sold or destroyed, but should be held until the manufacturer has been informed of the supposed defect. Felt manufacturers invariably make it a rule that felts claimed to be defective must be returned for their inspection.

88. Dryers.—The purpose of the dryers is to remove by means of heat the 65 per cent. or more of water left in the paper after it has passed through the presses. The dryer consists of a series of cast-iron cylinders usually erected so there is a double row of them, one on top of the other, and geared so they will all move at the same speed. Heat is supplied by steam pipes into these cylinders; the water of condensation is removed by various devices.

The dryer cylinders are of cast iron. The most ordinary size is 4 feet in diameter. The castings must be perfect, as the presence of flaws, sand holes, and other defects often found in large castings is dangerous, as a considerable amount of pressure is exerted on the dryers to provide the necessary degree of heat. The cylinders should be bored out smoothly so that the shell is of a uniform thickness throughout its circumference.

Drying cylinders should be turned on the outside and polished as bright and smooth as possible. The presence of tool marks or blemishes of any kind is fatal to the finish of the paper.

89. Arrangement of Dryers.—The most usual arrangement of the dryers is that already referred to, namely, in two rows, one above the other. With such an arrangement each tier usually has one long dryer felt. The majority of dryers in America are arranged in this way. European machine builders have inclined more toward separating the dryers into two, three, or more nests, each nest having an independent dryer felt.

Frequently the dryers are arranged in three tiers instead of two. This permits of some economy in space and also conserves heat to a certain extent.

90. Heating of Dryers.—The dryers must be piped in such a manner that the sheet of moist paper will not be scalded when it starts through the dryer. This is accomplished by having the first dryers next to the presses considerably cooler than those further along. In this way the temperature and the drying effect is gradually raised. There must be drying capacity enough to dry the paper at the required rate of speed without the exertion of undue force. If the sheet is seared or scalded at the beginning of the drying operation, it traps moisture in the top of the sheet, thus requiring a great deal more steam to dry it, besides destroying some of the desirable qualities of the paper.

91. Dryer Felts.—Dryer felts are among the most difficult of paper-machine accessories to manipulate. They are made of very hard and firm material; and they do not

stretch like woolen felts, except by wetting and drying. If by any means they become wrinkled, such wrinkles are usually permanent. All rolls and dryers over which these felts must run should be absolutely level and in line. Rolls, whether of wood, or iron, should be absolutely true.

Dryer felts should be equipped with an automatic tightener roll. They are subject to such a variety of conditions on account of the heat of the dryers, the moisture of the paper,

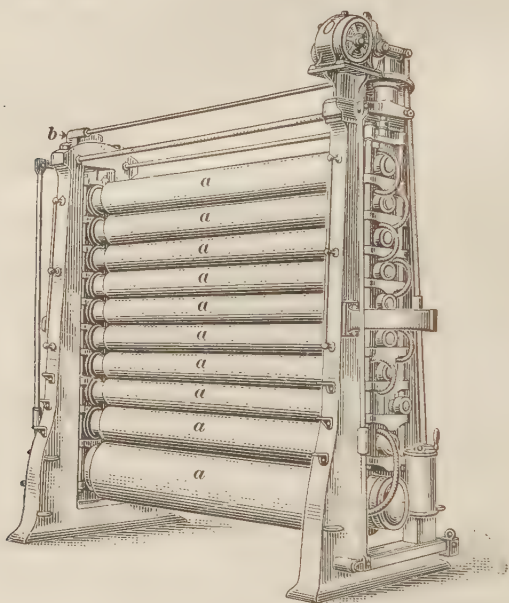


FIG. 13

and the speed of the machine, etc., that it is very necessary that the take-up roll to respond to all the variations should be very sensitive.

Carrying rolls not in proper alinement with the dryer felt are sure to cause the felt to travel from one side of the machine to the other, and in many cases to wrinkle. Corner rolls, especially, must be in perfect adjustment.

92. Tub Sizing.—When paper is to be animal-sized after it has passed over a couple of the drying cylinders,

it passes through a vat of liquid animal size and then between two rollers, which squeeze out the excess of size. The paper is then wound on a reel. This operation is omitted in the case of ordinary paper such as newsprint, wrapping, etc.

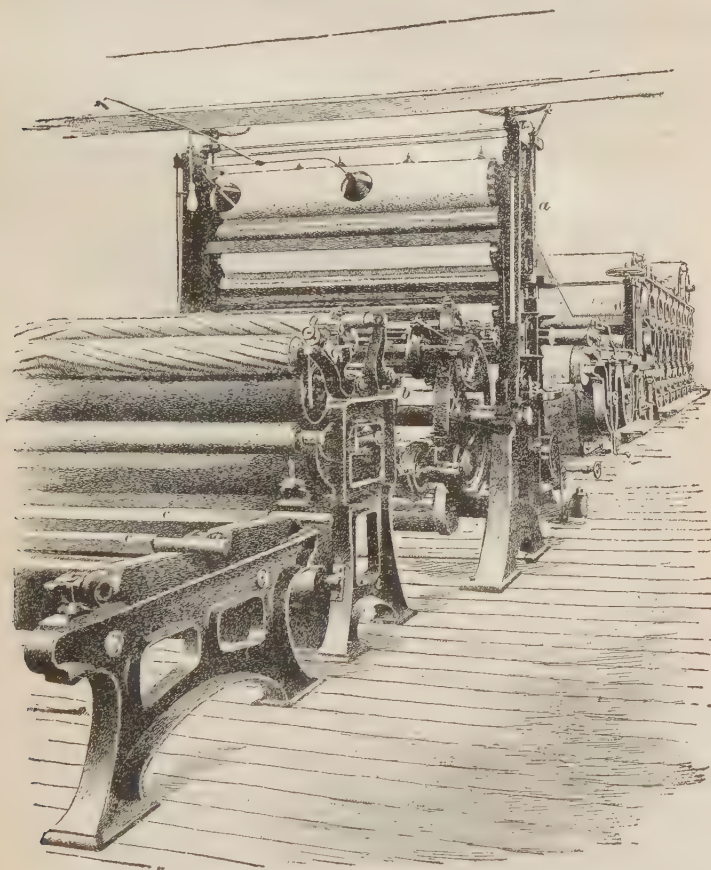


FIG. 14

In some mills, the paper is allowed to stand for a time and is then passed from the reels over a series of wooden drums furnished with fans, by means of which the paper is dried slowly. The most general custom is to pass the paper from the squeeze rolls over another series of dryers, and thus keep

the sheet in continual motion. After passing over the dryers, the sheet is transferred to the calenders.

93. Calendering.—In the operation known as calendering, the paper is given a high finish by pressing it between rolls on a machine called a *calender*. A calender, as shown in Fig. 13, consists of a series of highly polished, revolving, iron cylinders *a*, supported in a heavy frame that has a screw arrangement *b* at the top for regulating the pressure.

The number of cylinders on different machines varies, some having as high as three sets; the machine shown in Fig. 14

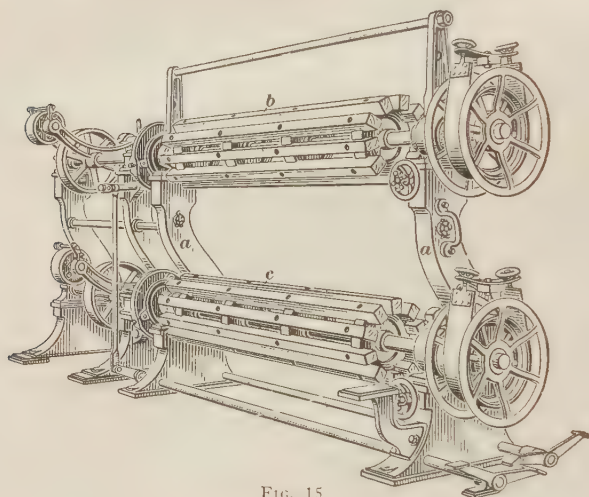


FIG. 15

has only one set *a*. The paper passes over these rolls, acquiring a high finish, and thence to the reels shown at *b*. In order to give a strong glaze to the surface of the paper, one or more of the calender rolls are kept hot by passing steam through them; and, again, in order to produce what is known as a water finish, some of these heated rolls are also kept wet.

94. Reels.—There are two different types of reels: stack reels and revolving reels. Stack reels shown in Fig. 15 are put one on top of the other in a vertical frame *a*—usually two reels *b* and *c* to a frame, sometimes three. The frames

are so constructed as to permit the reels being taken out, after they are filled, for the purpose of rewinding. After the reels are removed from the stack, they are placed on a separate set of stands for rewinding into smaller rolls or for cutting off into sheets.

Revolving reels shown in Fig. 16, consist of a set of reels arranged in the form of a cylinder. It is in fact a reel of reels. The housing carrying the reels revolves so that by the time one reel is filled another is in position to take its place, and, similarly, by the time one reel is almost unwound, another full reel

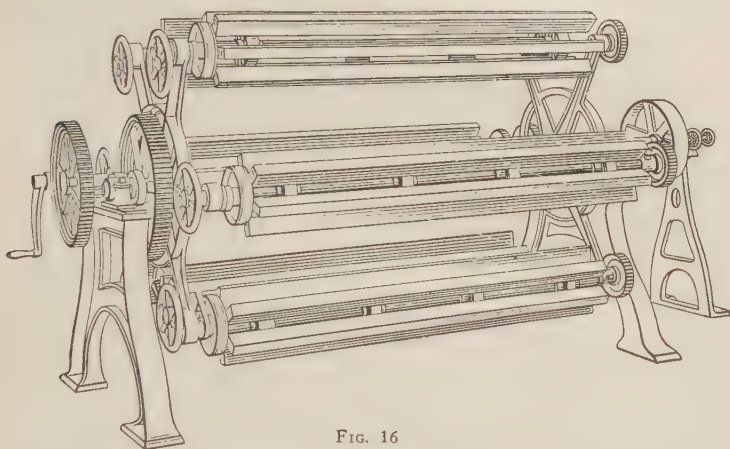


FIG. 16

is in position for unwinding. These reels are specially adapted where the paper is taken from the reel to be cut into sheets.

95. Stack reels are driven with a clutch and a friction belt is provided so the speed of the reel can be controlled, both in winding when care must be exercised not to break the paper, and in unwinding when friction must frequently be applied to keep the reel from going too fast and allowing the paper to become slack. The revolving or cylinder reels are driven by a gear arranged so that the gear for each reel meshes into the driving gear when the reel reaches a certain point in its revolution. These reels are also provided with frictions for the same purposes as in the case of the stack reels.

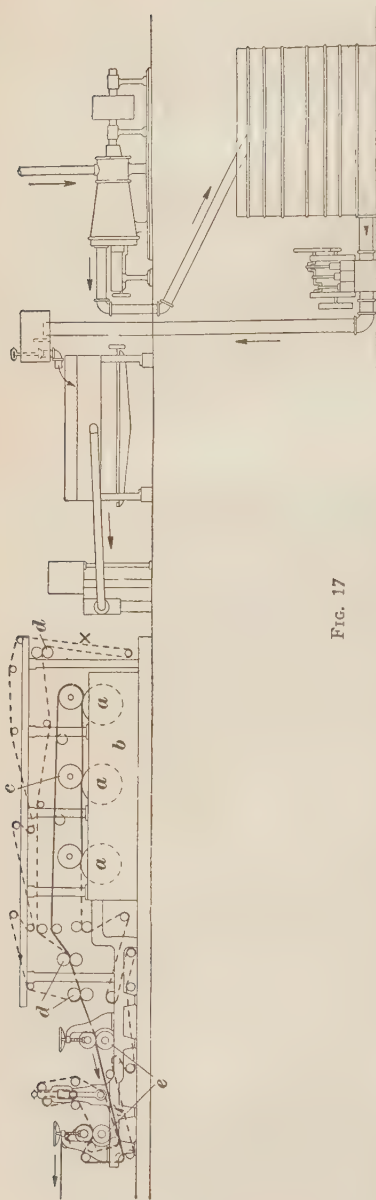


FIG. 17

96. Winders.—The winder is a machine for taking the paper from the reels and winding it in rolls of any desired size and at the same time cutting the paper into any desired width, which it does by means of knives that press on the paper as it is moving from the reel to the winder. The usual form of knife is a revolving one. These knives are known as *slitters*. Sometimes they are actuated by power and sometimes by the friction of the paper.

97. Cylinder Machine.—In Fig. 17 is illustrated the principle of the cylinder machine which consists of one or more cylinders *a* covered with wire, like the wire of the Fourdrinier machine, immersed in a vat of stock *b* in which they rotate. For the sake of simplicity, a single-cylinder machine will first be described. As the cylinder revolves in the vat, naturally the stock tends to flow through the wire and the fibers, being unable to pass, are caught in a web on the wire. The water that passes

through the wire is drained off from the end of the cylinder. The difference in level between the water in the cylinder and the stock in the vat, provides a suction that keeps forming a film on the wire as the cylinder moves around. Facing the head of the cylinder is a plate of the same size and shape as the head, and around the combined edge of the cylinder head and this plate is a band tight enough to preserve the suction from the cylinder, but not tight enough to interfere with the revolution of the cylinder. The amount of the suction and, consequently, the thickness of the layer of fibers formed on the cylinder is regulated by the height at which the water is allowed to stand in the cylinder. This is governed by a diaphragm controlling the opening by which the water drains away from the cylinder.

The long straight fibers are naturally drawn head-on against the cylinder wire, like logs going down stream, and, to prevent this action, which would not give as good a sheet as if the fibers felted and matted more on the screen, various devices are inserted in the vat to keep the stock in motion so that the fibers will be compelled to pass the surface of the screen with their length parallel to it to a greater or less extent, and, consequently, be pulled against it by the suction sidewise.

At the top of the cylinder is a couch roll *c* over which passes a felt moving in the same direction as the surface of the cylinder. This felt picks up the film of fibers and carries it along through one or two pairs of squeeze rolls *d* to the presses *e* and dryers which are arranged like those of the Fourdrinier machine.

For making heavy boards, which consist of layers of several different kinds of paper, after the felt has passed over the first cylinder it may pass over a second cylinder, revolving in a vat, receiving another layer of paper, and in the same manner a third and fourth—as many as eight sometimes being used.

98. Harper-Fourdrinier Machine.—The Harper-Fourdrinier machine closely resembles an ordinary Fourdrinier machine with the entire portion preceding the presses turned around end for end. In other words, the wire is traveling away from the presses, instead of toward them. The paper

formed on the wire is carried back from the couch rolls on a long felt (which is carried on rolls high over the wire) which supports the paper until it enters the presses and sometimes even until it enters the dryers.

The chief usefulness of this machine lies in the fact that the paper is constantly supported by a felt, or some other surface, there being no gaps to bridge, as between the couch rolls and presses in the ordinary Fourdrinier machine. This renders the Harper machine valuable for making very delicate papers such as tissues, cigarette paper, crêpe papers, etc. Since this kind of paper requires very little pressing, there is frequently only one set of press rolls on such a machine. The excessively long felt, often 100 feet in length, is one of the undesirable features of this machine. In the first place, it is very expensive and, secondly, it is very hard to keep free from injury. This fact, together with the up-keep of the Fourdrinier wire, makes the Harper a very expensive machine to maintain. Consequently, its use is restricted to those kinds of paper that cannot well be made on any other machine.

99. Yankee Machine.—The essential difference between a Yankee machine and a Fourdrinier, cylinder or Harper, is the method used in finishing or surfacing and drying a sheet of paper. The Yankee machine has one very large dryer, sometimes considerably more than 10 feet in diameter, while the ordinary machine dryers range from 3 feet to 5 feet in diameter. The large dryer which is used on Yankee machines has a very highly polished surface, and against this surface a set of press rolls run, the top press roll coming in contact with the surface of the dryer. The press rolls are permanently fixed and the dryer is screwed back against the surface of the press roll by means of screw gears and hand wheels. The reason is that when the paper passes between the rubber-covered press rolls and dryer, it can be pinched very hard. A dryer felt also usually covers the big Yankee dryer the same as it covers ordinary dryers, care being taken to get all of the drying surface possible within the radius of the dryer.

The making-up part of the machine is precisely the same as that of any other machine, such as the straight Fourdrinier part, wet part of cylinder machine or wet part of Harper-Fourdrinier machine. Sometimes there are small intermediate dryers and the big Yankee dryer is placed at some advantageous point in the section of dryers. The object of all of this is to give the sheet of paper a glossy finish on one side only. The production of the machine is limited to the capacity of the dryer. This machine gives an excellent shiny surface to the paper which cannot be obtained by ordinary calendering, but it only gives the surface to one side of the sheet, the sheet of paper being hugged so tightly against this dryer by the dryer felt and moved so slowly that by the time it goes over the dryer once, it is ready to reel up nicely finished.

100. There are a great many uses for this paper, such as for the lining of duplex paper bags with the shiny side inward. It makes a very satisfactory appearance for a bag containing cereals of any kind, coffee, teas, confectionery, etc. Tissue paper is sometimes finished in this way, especially that used for wrapping confectionery goods. It is also used for paper for druggists' purposes. It may be used for blank leaves in technical books, novels, etc., where a nice finish is desired. Paper napkins and towels and all sorts of paper used for similar purposes may be made and finished on a Yankee machine, which is adapted for thin and medium weight papers only, ranging from tissues to not thicker than 35- or 40-pound paper. The reason for this is also on account of the limited production and slow running of such a machine, because all of the finish that it can possibly get is gained while the big dryer is making one revolution.

101. Supercalendering.—It is sometimes necessary to have an extra-high finish on the paper. This is done on glazing calenders, which are generally called *supercalenders*. These machines are also of various types, one being a series of rolls that are alternately of highly polished iron and compressed paper, the iron rolls being hollow and heated by steam. Another type consists of a stack of highly polished,

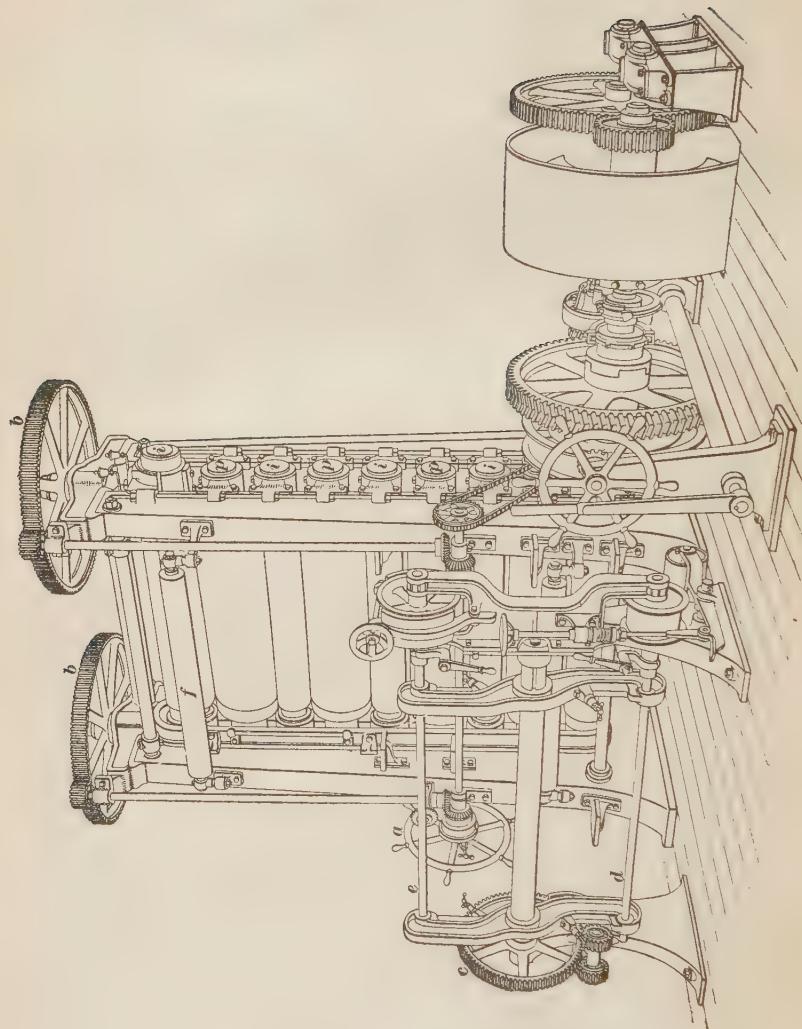


FIG. 18

chilled-iron rolls. The rolls are bored out and can be heated by steam. There is also an arrangement by which one or more of the rolls can be raised, according to the finish required.

The supercalender in most common use is shown in Fig. 18. It consists of a stack of rolls that are alternately of highly polished iron and compressed paper, neither of which is heated by steam. These rolls are pressed down against each other by turning a wheel *a*, which operates the large wheels *b*, at the top of the calenders. The lower roll is made to revolve at a high speed, and by means of the friction exerted on the second roll, it revolves in the opposite direction, and, in a like manner, all the other rolls revolve at a high speed. Owing to the great friction on these rolls, they become very hot. The iron and paper rolls are designated by the letters *i* and *p*, respectively.

102. The roll of paper to be calendered is transferred from a small truck to the bar *d*, when, by turning a crank at *c*, the position of the roll is changed to *e*. The sheet is then passed up to a machine tender, who passes it under a brass roll *f* and then around the rolls of the calender, finally winding it on a core placed on the rod *d*. It is customary to have a perforated steam pipe extending across each side of the machine, so that a little steam can strike both sides of the sheet as it starts over the calenders. The paper coming from the calenders has a very high finish, and is transferred to the slitting and rewinding machine.

FINISHING OF PAPER

103. Final Processes.—After the paper is formed into large rolls (usually called *jumbo rolls*) on the reels at the end of the paper machine, it is sent to a department of the mill known as the *finishing room*, where it is further prepared for shipment and sale. Finishing-room operations fall into two general classes: (1) Making rolls of paper; (2) making bundles of paper. Whether rolls or bundles of paper are made, depends entirely on the purpose for which the paper is to be used.

In a newsprint mill the work of finishing is very simple, the paper being sold in large rolls, practically as it comes from the machines. In a mill making fine writing or book papers there are a great many operations to be carried out in the finishing room, which then becomes one of the most important parts of the plant.

104. Making Rolls of Paper.—The paper, as it is wound off the jumbo rolls formed on the reels in the machine room, passes onto a *rewinder*, with which is combined a *slitter*, so that as the paper is rewound it is cut lengthwise into rolls as may be required.

Simple as this operation sounds, it actually requires the most careful attention. The adjustment of the slitter knives must be perfect, otherwise oversized and undersized rolls will result. The roll must be held absolutely straight, and the rolls must be rewound in a uniform manner. Otherwise rolls would be formed having hard and soft spots. Defective rolls of this kind are practically useless when the paper is to be used in any kind of automatic machinery such as modern paper-bag-making machines, cigarette machines, high-speed printing presses, etc. Thus, it is seen that there is every opportunity to ruin otherwise perfect paper in a badly managed finishing room.

There are two methods of rewinding: (1) The center-rewind method; (2) the surface-rewind method. The center-rewind method calls for the application of power to the rewind shaft, which extends through the center of the roll of paper that is forming on it. Trouble is experienced owing to variation of thickness in the paper across the width of the sheet, causing the rolls to build up larger where the web is thicker, which pulls the paper faster on the thicker portions, causing the web to feed unevenly. Moreover, when narrow rolls are required, they will not stand alone after they have been built up to any considerable diameter.

The center-rewind method has been abandoned, in most modern establishments, in favor of the surface-rewind method. By this method all the rolls are formed side by side on a single

shaft, by surface contact with a pair of revolving support rolls, on which the paper rests. No power is applied directly to the rewind shaft. This method is entirely satisfactory when properly operated.

Slitting is carried out either with the *rotary shear* or *slitter*, or with the *score cutter*, the latter being more modern and efficient. The rotary slitter is shown in Fig. 19. It consists of a number of circular knives *a*, mounted on a rigid shaft *b*, and cutting against the web of paper *c* as it rolls

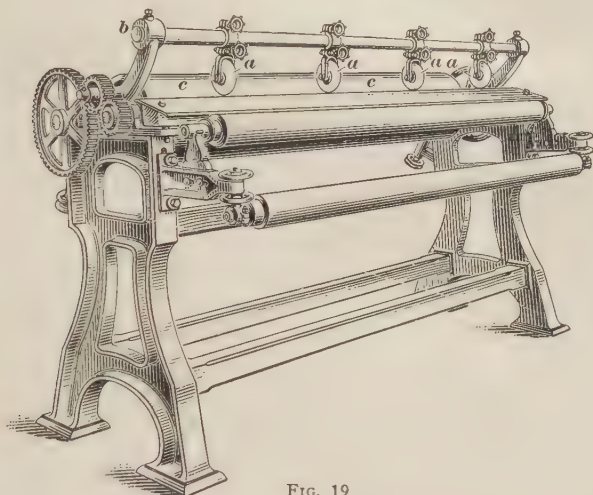


FIG. 19

up on the rewinder. Unless the rotary knives are perfectly sharp, they cause frayed edges on the paper, which interlace and cause trouble in separating the rolls.

The score cutter, shown in Fig. 20, consists of cutting disks *a* with **V** edges so mounted as to revolve under spring pressure against the surface of a glass-hard steel cylinder *b*, the surface of which is very smooth and highly polished. The paper enters the machine at *c* and is wound up after slitting on the shaft *d*, forming rolls as at *e*. The machine is driven by a motor at *f*. The steel cutting wheel is mounted with ball bearings and revolves practically without friction. This appliance invariably yields a clean-cut, even, perfect roll or sheet of paper, even

when operated at high speeds, under which conditions the rotary slitter gives a lot of trouble.

Plugs are inserted in the rolls as soon as they come off the slitting and winding machinery, so that when they are piled up for transportation or storage they will not lose their

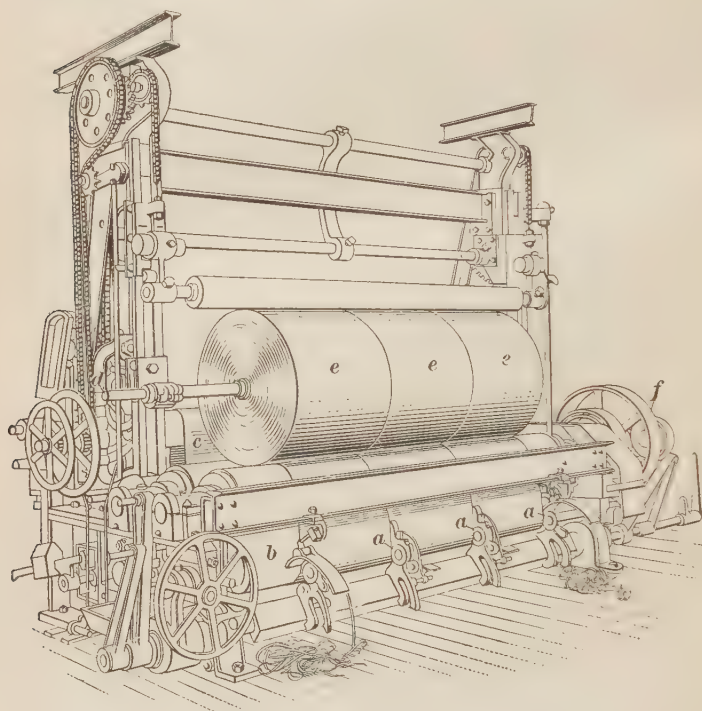


FIG. 20

symmetrical cylindrical shape. These plugs are simply small blocks of wood cut to the proper shape and size.

The roll is wrapped with several plies of some good durable paper, which is folded over at the end and pasted down with a label, usually printed with the name and trade-mark of the mill. An inside header should always be inserted before turning down the ends of the wrapper, so as to prevent paste from the label from getting into the end of the roll, causing the paper to stick together on the edges.

105. Making Bundles of Paper.—The finer sorts of paper are shipped in flat bundles. This kind of finishing does not require as intricate machinery as roll finishing, but it requires more labor. The jumbo rolls from the paper machines are placed on an unwinder, from which they are fed to the cutters, where they are cut into the desired widths, and also cut off transversely at regular intervals, thus making rectangular sheets of regular and exact size. The sheets are gathered up, counted, and placed in piles. In some mills this is still done by hand, but machines called *lay boys* are mostly used for this purpose.

The bundles of sheets are wrapped, or boxed in wooden cases, and placed in storage. Sometimes very large sheets are folded before being wrapped.

MANUFACTURE OF PAPER

(PART 3)

TESTING AND ANALYSIS OF MATERIALS AND FINISHED PRODUCTS

APPARATUS AND CHEMICALS

1. This Section will deal with the tests and analyses of the chemicals, liquors, etc., used in the manufacture of paper; with the testing of pulp and of finished paper; and with various methods of making quick but tolerably accurate tests by means of which the manufacturer may be able to operate his plant on an efficient and economical basis. The size of the laboratory and the extent of the equipment required depends very largely on the size and scope of the manufacturing processes carried on in the pulp and paper mill.

The equipment described is the minimum laboratory installation that would be satisfactory for any work of real value in a modern pulp and paper plant. It is important that a pulp- and paper-mill chemist should have at hand, and kept in good order, all the apparatus and chemicals that are likely to be required at any time. Therefore, in order to give an idea of what will be essential in even the smallest laboratory, a list of apparatus and chemicals necessary for this purpose is here given.

2. **Apparatus.**—The following apparatus will be found necessary for the analytical work about to be described: A sensitive balance enclosed in glass case, the interior of which should be well desiccated, provided with a rider beam, divided into tenths of milligrams, and a set of weights ranging from 1

milligram to 100 grams. Whenever possible, the balance should be set up in a clean, dry room, separated from the laboratory proper by a wall, or partition. Where this is not possible, it is advisable to enclose the balance in a small booth. If the building in which the laboratory is situated is subject to vibrations from heavy machinery, it may be necessary to build a brick or concrete pier reaching well into the ground, for the support of the balance. One large and one small desiccator; several air-tight boxes with screw lids, for taking pulp samples (Mason jars can also be used for this); one large steam bath (which should be connected with a still;) one hot-air bath and one water bath for slow evaporation; porcelain dishes, sizes 4 and 6 inches; measuring flasks of the following capacities: 20, 50, 100, 250, 500, and 1,000 cubic centimeters, when filled to the mark on the neck; plain ungraduated flasks of the following capacities: 100, 250, 500, and 1,000 cubic centimeters; several 50-cubic-centimeter burettes, graduated into cubic centimeters and tenths of cubic centimeters, each provided with a stand; a set of pipettes, including the following sizes: 1, 2, 5, 10, 25, 50, and 100 cubic centimeters; a set of graduated cylinders of the following capacities: 100, 500, and 1,000 cubic centimeters (preferably glass-stoppered for mixing solutions); several ungraduated cylinders, each having a capacity of about 200 cubic centimeters; beakers, lipped shape, from Nos. 1 to 6; watch glasses of various sizes and a pair of ground glasses and clips; one iron, one large porcelain, one small porcelain, and one agate mortar, with pestles; funnels of various sizes; a separatory funnel; a Gooch filter; glass and rubber tubing, various sizes; a platinum dish having a capacity of 50 cubic centimeters; platinum crucibles, 15 and 20 grams capacity, respectively; platinum triangles for crucibles, and light and heavy platinum wire; ring stands and Bunsen burners; test tubes, various sizes; porcelain crucibles, various sizes; alundum cones; a filtering stand; a set of reagent bottles; sample bottles, various sizes; several liter bottles for standard solutions; a wash bottle; a drying apparatus and train for CO_2 determinations; a Jones reductor; thermometers, 212° F. and 212° C.; short-stem Baumé hydrometers of the following

ranges: 3° to 4° , 0° to 5° , 0° to 8° , 0° to 15° , and 15° to 40° ; stirring rods, forceps, spatulas, and pinch cocks.

3. Hydrometers.—The hydrometer, whether it be on a Twaddell or a Baumé scale, is very useful about a paper mill. This instrument is used in all departments of the mill for the efficient regulation of the strengths of solutions. The reading of the hydrometer is affected by the temperature, an increase in temperature decreasing the density of solutions. For this reason it is customary to add 1° Baumé for each 30° of tem-

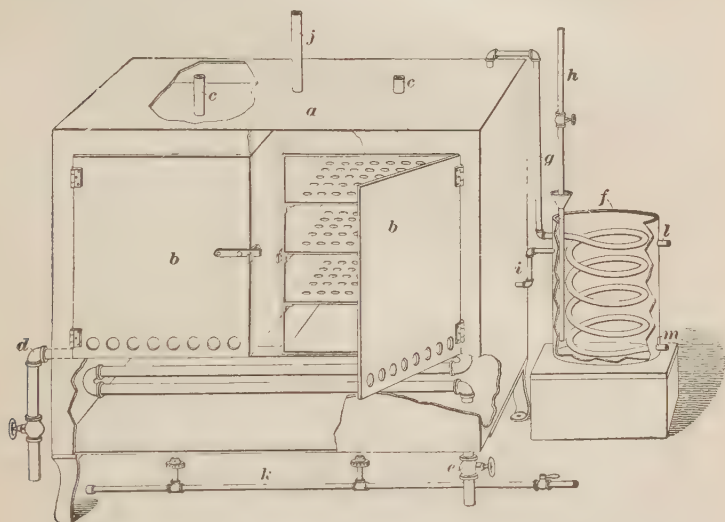


FIG. 1

perature over 60° F. This allowance, however, will hold good only for temperatures up to about 150° F. A greater allowance should be made for temperatures above this, about $4\frac{1}{2}^{\circ}$ Baumé being allowed for a temperature of about 185° F.

4. Drying Ovens.—A good drying oven is essential in the pulp- and paper-mill laboratory, not only for determining moisture in paper and in pulp, but also for the analysis of clay, soda ash, and many other materials. There are many different kinds of drying ovens, the most modern, efficient, and convenient of which are heated with electricity. In spite of its

higher first cost, an electric oven should be installed wherever electricity is available. They permit of very accurate adjustment of the temperature. The temperature can be maintained anywhere between room temperature and 260° C., accurate to within a fraction of a degree. There is no fire risk and they do not heat up the laboratory.

Where it is not possible to have an electrically heated oven, the next best device is a steam-heated oven. The bath shown in Fig. 1 has proved a very convenient form of steam-heated drying oven. It consists of a large copper box *a*, which has two drying compartments *b*, closed by means of doors perforated at the bottom, each having a chimney *c* extending to the outside, to permit the free passage of air through the bath. In each compartment there are perforated shelves, which can be removed if desired. The compartments are so arranged that steam circulates all around them. The bath is connected with the still *f*, which serves as a feed for it by means of the small connecting pipe *i*, the water in the bath being kept at a constant level with the overflow pipe *l* of the still. The pipe *g* comes from the top of the bath and connects with the worm of the still, the distilled water escaping at *m*. The cooling water is furnished to the still through pipe *h*. The still is at such a height that the bath is always half full of water, which is kept at a slow boil by means of the steam coil, which enters at *d* and passes out at *e*. A pipe *j* extends into the bath between the two compartments, and is provided with a valve, which, when open, stops the preparation of distilled water. As a precaution against the steam being turned off, a gas line *k* provided with two burners is run under the bath.

5. Freas Electric Conditioning Oven.—The Freas electric conditioning oven is specially designed for determining moisture in pulp and paper, and also in textile fabrics and other materials. It consists of an electrically heated oven automatically kept at the desired temperature by an *all-metal thermostat*. Temperatures up to 130° C. can be maintained indefinitely. The oven is equipped with baskets to hold the samples to be conditioned, and is an ingenious arrangement whereby the

samples can be weighed in the oven after conditioning, without being exposed to the exterior atmosphere. A balance is provided for weighing the samples before and after drying, either the torsion balance or an analytical balance. A motor-driven fan provides a circulating atmosphere and forces the withdrawal of the moisture-laden air, thereby reducing to a minimum the time required for drying. The oven is mounted on an iron base, which stands on a table or bench.

6. Chemicals.—All chemicals used should be chemically pure, or what is known as *analyzed chemicals*. The following is a list of the principal chemicals necessary for all analytical tests given in the following pages:

Hydrochloric acid	Barium hydroxide
Sulphuric acid	Barium chloride
Nitric acid	Barium peroxide
Acetic acid	Bromine
Oxalic acid	Calcium chloride
Arsenious acid	Ether
Sulphanilic acid	Carbon bisulphide
Alcohol	Iron perchloride
Aniline sulphate	Iron protosulphate
Ammonium oxalate	Iron piano wire
Ammonium phosphate	Iron sulphide
Ammonium sulphite	Lead acetate
Ammonium chloride	Lead peroxide
Ammonium sulphocyanide	Mercury
Methyl orange	Sodium sulphate
Manganese chloride	Sodium phosphate
Marble chips	Sodium carbonate, dry
Phenol	Sodium chloride
Platinic chloride	Sodium sulphite
Phloroglucine	Sodium hydroxide
Phenolphthalein	Sodium acetate
Potassium iodide	Sodium nitrate
Potassium ferrocyanide	Soda lime
Potassium permanganate	Silver nitrate
Potassium bisulphate	Starch
Potassium chromate	Zinc sulphate
Potassium sulphate	Zinc chloride
Potassium sulphocyanide	Zinc oxide
Potassium chlorate	Zinc shot
Potassium hydroxide	

7. Standard Solutions.—The following standard solutions will be found necessary:

Normal sulphuric acid	Normal sodium carbonate
Normal hydrochloric acid	<i>n</i> /10 sulphuric acid
Normal oxalic acid	<i>n</i> /10 sodium hydroxide
<i>n</i> /10 iodine	<i>n</i> /10 sodium arsenite
<i>n</i> /10 sodium chloride	Standard potassium permanganate
<i>n</i> /10 silver nitrate	<i>n</i> /20 silver nitrate solution
Normal sodium hydroxide	<i>n</i> /20 potassium sulphocyanide

8. The preceding standard solutions used in volumetric analysis are made up as follows:

Normal sulphuric acid is prepared by mixing 30 cubic centimeters of pure sulphuric acid of 1.84 specific gravity with 150 cubic centimeters of distilled water in a 1,000-cubic-centimeter measuring flask allowing the mixture to cool to room temperature. Dilute to 1,000 cubic centimeters with distilled water and adjust the volume by titrating against 5.3 grams of pure, dry sodium carbonate dissolved in about 100 cubic centimeters of water until 100 cubic centimeters of the acid will just neutralize the 5.3 grams of sodium carbonate. This titration should be carried out according to the directions subsequently given for the determination of total alkali in soda ash.

*Tenth normal (*n*/10) sulphuric acid* can be prepared by measuring 100 cubic centimeters of the normal sulphuric acid, in an accurately graduated pipette that has been well cleaned and rinsed with the acid. The contents of the pipette are carefully rinsed into a liter flask and diluted to the mark.

Normal sodium carbonate is prepared by weighing out about 53 grams of dry sodium carbonate, dissolving in about 500 cubic centimeters of hot water, after which it is cooled, transferred to a liter flask, and diluted to the mark. This should be matched against the normal acid, using methyl orange as an indicator.

Normal sodium hydroxide should contain 40 grams per liter of sodium hydroxide. Dissolve 41 grams of chemically pure sodium hydroxide in 250 cubic centimeters of distilled water in a 1,000-cubic-centimeter measuring flask. Allow to cool to room temperature and dilute to 1,000 cubic centimeters.

Adjust by adding distilled water until 50 cubic centimeters are exactly neutralized by 50 cubic centimeters of normal sulphuric acid, titrating cold with 5 drops of phenolphthalein indicator.

Tenth normal ($n/10$) sodium hydroxide can be made by carefully measuring 100 cubic centimeters of the normal solution and diluting to 1 liter. This can be verified by matching against $n/10$ sulphuric acid.

Normal hydrochloric acid is prepared by taking 100 cubic centimeters of HCl (1.2 specific gravity) diluting to 1 liter, and matching against normal soda solution, using methyl orange as an indicator. This acid can be further rectified by precipitating with silver nitrate and calculating the number of cubic centimeters necessary to make 1 liter of strictly normal acid.

Normal oxalic acid is made by dissolving 63 grams of pure crystals of oxalic acid in distilled water and diluting to almost 1 liter. This normal solution can be rectified by matching against a strictly normal alkali. It should be kept in a dark place.

Tenth normal ($n/10$) sodium arsenite solution is prepared by dissolving 50 grams of sodium bicarbonate in about 200 cubic centimeters of hot water and adding 4.95 grams of chemically pure, dry, powdered, arsenious acid, As_2O_3 . Arsenic trioxide of the required purity is difficult to obtain, and only that specially prepared for analytical work should be used. Moreover, it should be thoroughly dried before weighing. Cover the beaker with a watch glass and allow the contents to remain almost at a boil, stirring from time to time, until all the arsenious acid is dissolved. Cool, transfer to a liter flask, and dilute to the mark.

Arsenious acid dissolves more readily in the normal carbonate, but owing to secondary reactions, which interfere with the reaction between sodium arsenite and iodine, its use is not advisable.

Sodium-arsenite solution is very unstable and needs to be restandardized frequently, which can be done by matching it against $n/10$ iodine solution, using starch as an indicator.

Tenth normal ($n/10$) iodine solution is prepared by dissolving 12.7 grams of iodine with 18 grams of potassium iodide in 100 cubic centimeters of distilled water. If the potassium iodide is dissolved in a small amount of water, as directed, and the iodine then added, solution will take place very readily. The solution should not be heated. When dissolved, the iodine solution is transferred to a liter flask and diluted almost to the mark. In order to rectify this $n/10$ solution, it is matched against the $n/10$ sodium arsenite, using starch as an indicator.

Standard potassium-permanganate solution is prepared by dissolving about 3.25 grams of chemically pure, crystalline, potassium permanganate in about 200 cubic centimeters of distilled water and filtering this solution through glass wool or a Gooch filter provided with an asbestos mat. Dilute to approximately 1,000 cubic centimeters and place in a dark-colored bottle away from direct sunlight. The solution can be standardized in a number of different ways of which the following is probably the most accurate:

Weigh out with the utmost accuracy 6.7 grams of pure, freshly dried sodium oxalate and dissolve it in about 300 cubic centimeters of hot, distilled water. Place this solution in a 1-liter flask and allow it to cool to exactly 20° C. Dilute with distilled water at 20° C to exactly 1 liter. This solution will be exactly $n/10$. Measure out about 50 cubic centimeters of the above solution into a flask and 15 cubic centimeters of pure concentrated sulphuric acid and heat to boiling. Titrate this with the potassium-permanganate solution, adding the latter drop by drop at first, stopping at the very first appearance of a faint but permanent pink color. A number of these titrations should be run for the sake of accuracy. To obtain the factor of the potassium-permanganate solution, divide 50 cubic centimeters by the number of cubic centimeters of permanganate solution required for the titrating.

Standard size solution is prepared as follows: Dissolve 20 to 25 grams of good, heavy, rosin size in about 250 cubic centimeters of 95-per-cent. alcohol, filter and dilute almost to 1 liter with a 60-per-cent. alcohol solution. Add a little phenolphthalein and then add a solution of caustic soda,

drop by drop, from a burette, shaking after each addition, until a faint pink color appears. This neutral size solution is used as a standard after first determining its value. The value is determined by means of pure, crystallized, potassium alum, 1 part of which precipitates 2.36 parts of neutral rosin size. Weigh out 10 grams of the alum crystals that have previously been pressed between two filter papers to remove any adhering moisture. Dissolve and dilute to 1 liter. One cubic centimeter will then contain .01 gram of alum. A flask with a capacity of about 200 cubic centimeters is then filled about two-thirds full of distilled water, and 20 cubic centimeters of the size solution is run in from a burette. The alum solution is then run in from another burette, a few drops at a time. The flask is then closed with a stopper, shaken vigorously, and allowed to stand after each addition, until the precipitate rises to the top, which it will do very rapidly. This is continued until the solution is left perfectly clear, after the precipitate has risen. The number of cubic centimeters of alum used multiplied by .01 will give the amount of alum, in grams, required to precipitate 20 cubic centimeters of standard size. This, multiplied by 2.36, will give the number of grams of neutral size in 20 cubic centimeters of this solution. If 10 cubic centimeters of the alum solution is used, 20 cubic centimeters of the standard size will contain .236 gram of neutral rosin size.

Tenth normal ($n/10$) sodium-chloride solution is prepared by accurately weighing 5.837 grams of chemically pure sodium chloride (which has been previously heated in a covered platinum crucible to a dull-red heat for several minutes, and cooled in a desiccator), dissolving in distilled water, and making up to 1 liter. It is always advisable to keep this standard solution on hand to assist in making up $n/10$ silver-nitrate solutions.

Tenth normal ($n/10$) silver-nitrate solution is prepared by weighing out 17 grams of pure crystallized silver nitrate, dissolving and diluting to almost 1 liter. This solution is then matched against the $n/10$ sodium-chloride solution, using potassium as an indicator, and adjusted to an exact $n/10$ solution.

One cubic centimeter of this silver-nitrate solution will then be equivalent to .00355 gram of chlorine or .00585 gram of sodium chloride.

Twentieth normal (n/20) silver-nitrate solution can be prepared by dissolving 8.5 grams of silver nitrate in distilled water, to which a drop of colorless nitric acid of 1.42 specific gravity has been added and diluting to 1,000 cubic centimeters with distilled water. This solution is practically *n/20* and can be standardized in exactly the same manner as the *n/10* silver-nitrate solution.

Twentieth normal (n/20) potassium sulphocyanide solution should contain 4.86 grams per liter of potassium sulphocyanide. Dissolve 4.86 grams of chemically pure potassium sulphocyanide in 500 cubic centimeters of distilled water and make up to 1,000 cubic centimeters with distilled water. It should be adjusted to exactly equal the *n/20* silver-nitrate solution by titrating against 10 cubic centimeters of the *n/20* silver-nitrate solution to which 2 cubic centimeters of the iron indicator have been added, until a permanent pink color is obtained.

9. Indicators.—The following indicators will be found necessary in making analyses of the raw materials that enter into the manufacture of paper:

Phenolphthalein indicator is best prepared by taking 105 cubic centimeters of ethyl alcohol (95 per cent.) and in this dissolving 2 grams of phenolphthalein. When the phenolphthalein is thoroughly dissolved, add 95 cubic centimeters of distilled water. A few drops of this indicator will be sufficient for titrating; it will produce no color in acid liquids, but the slightest trace of caustic alkali will change it to purple red. This indicator can be used in alcoholic solutions and is useful when titrating organic acids, but it is not reliable in the presence of carbonates in a cold solution.

Methyl orange containing impurities that are not soluble in water should be recrystallized from alcohol. This indicator should not be used when titrating organic acids. A convenient strength is .1 gram to 100 cubic centimeters of water. One or two drops are sufficient for 100 to 150 cubic centimeters

of the solution to be titrated. A good end point cannot be obtained if too much of the indicator is used. This indicator gives a very sharp end point in the presence of carbonates, and is therefore very useful in the titration of the alkali carbonates.

Starch is prepared as follows: About 300 cubic centimeters of water is brought to a boil in a flask having a capacity of about 1 liter. Four grams of wheat starch is then mixed in a little cold water until all the lumps are broken and a thin, pasty mass is formed; this is added to the boiling water, and the whole allowed to boil for 5 minutes. A mixture of 5 grams of zinc chloride in a little water is then added, and the whole allowed to boil 1 minute longer. This mixture is then removed from the flame, diluted to about 900 cubic centimeters, well mixed, and allowed to stand overnight. The clear starch solution is decanted into a bottle and is then ready for use.

Iodized starch is made by taking some of the starch solution just mentioned and adding a small amount of a solution of potassium iodide.

Potassium chromate of a convenient strength is made by adding 1 gram to 10 cubic centimeters of water. A few drops of the solution will be sufficient for 100 or 150 cubic centimeters of the solution to be titrated.

ANALYTICAL METHODS

DETERMINATION OF CHEMICALS

10. Analysis of Soda Ash.—The most necessary determination made on soda ash is the *total alkali*, or the sodium oxide. If the soda ash is the product of a reliable manufacturer and is fresh, it is usually not necessary to make any other determination. However, on exposure to air, soda ash takes up water and carbon dioxide, forming sodium bicarbonate. Consequently, if accurate figures are desired, it is always necessary to determine the bicarbonate on ash which has been exposed to the atmosphere for any length of time. Further-

more, if it is desired to determine the quality of the ash as originally shipped, such exposed ash should be dried for 2 hours at 200° C. before analysis.

The material should be carefully sampled, a sample being taken from each of at least six containers, selected at random, and thoroughly mixed. This sample should then be quartered down until a composite sample of convenient size is obtained.

In America, soda ash is always sold on the New York and Liverpool test, which is based on the erroneous atomic weight of sodium (24). When getting the actual alkali, it is advisable, also, to report it in terms of the New York and Liverpool test, by multiplying actual alkali found by 1.032. In England, soda ash is sold on the Newcastle test,* for which the actual alkali found should be multiplied by 1.013. Soda is sold on a guarantee of 58 per cent. Na_2O , and many chemists have rejected cars that have fallen below this test, to find later that the soda had been bought on a guarantee of 58 per cent. according to the New York and Liverpool test.

Determination of Sodium Oxide.—Dissolve 5 grams of the sample in 50 cubic centimeters of distilled water in a 500-cubic-centimeter beaker. With the beaker nearly covered with a watch glass to avoid mechanical loss, run in 93 cubic centimeters of normal sulphuric acid; boil off the liberated carbon dioxide, cool, and add 2 drops of methyl-orange indicator and titrate to the exact end point. In this titration, as the end point is approached, care should be taken to stir well, so that the color change from yellow to pink will be sharp and easily noticeable, the acid being added a drop at a time.

Cubic centimeters $n/H_2SO_4 \times .62 =$ the per cent. of Na_2O
(actual)

*Under the Newcastle test the actual alkali is calculated by an incorrect chemical equivalent for sodium oxide and is stated as 32/54, or 64/108, of the total carbonate of soda. This error originated in the fact that the early chemists fixed the atomic weight of sodium at 24, the correct value being 23.

Under the New York and Liverpool test, the incorrect chemical equivalent for sodium oxide (Na_2O) is used, while the correct equivalent for carbonate of soda is used. This test, accordingly, figures the actual alkali as 32/53, or 64/106, of the total carbonate of soda.

Cubic centimeters of $n/\text{NaOH} \times 1.68 =$ the per cent. of
 NaHCO_3

(New York and Liverpool test)

Determination of Sodium Bicarbonate.—Dissolve 5 grams of the sample in 50 cubic centimeters of distilled water in a 200-cubic-centimeter beaker. Titrate with normal sodium-hydroxide solution until a drop of the solution added to a drop of the silver-nitrate indicator on a spot plate instantly gives a dark color.

Cubic centimeters of $n/\text{NaOH} \times 1.68 =$ the per cent. of
 NaHCO_3

Determination of Sodium Carbonate.—To get the sodium-carbonate content, multiply the number of cubic centimeters of normal sulphuric acid used in the determination of sodium oxide by 1.06. The result gives the percentage of sodium carbonate, and, as previously mentioned, if the ash has been exposed to air, it is necessary first to determine the bicarbonate before the carbonate can be calculated.

Determination of Sodium Chloride.—Dissolve 2 grams of the sample in 50 cubic centimeters of distilled water in a 200-cubic-centimeter beaker. Add 5 cubic centimeters of nitric acid of 1.42 specific gravity and about 2 cubic centimeters of iron-indicator solution. Run in a few drops of $n/20$ potassium sulphocyanide solution from the burette, noting the quantity. Then titrate with $n/20$ silver nitrate, adding the silver-nitrate solution drop by drop and stirring constantly to decoloration. Add about .5 cubic centimeter more of the silver-nitrate solution, and filter off the silver chloride formed, washing well on the filter with distilled water. Titrate the combined filtrate and washings to a permanent pink color with $n/20$ potassium sulphocyanide.

(Cubic centimeters $n/20 \text{ AgNO}_3$ —cubic centimeters $n/20$
 $\text{KCNS}) \times .146 =$ the per cent. of NaCl

Only the above determinations are necessary in the case of soda ash purchased from reliable manufacturers. In fact, usually, the simple determination of total alkali, or sodium oxide, will be sufficient. If there is any suspicion that the

soda ash contains other impurities, it may be tested qualitatively for sulphates, iron, and alumina.

Table I gives the chemical and commercial equivalents for the different kinds of alkali. On the continent of Europe, alkali is sold by its strength in carbonate of soda (Na_2CO_3), according to column No. 1 of table. In England, alkali is sold nominally on its strength in actual alkali (Na_2O), as per column No. 2 of table, but actually on the so-called Newcastle test of the actual alkali, as per column No. 3 of table. In the United States, the commercial standard for 75 years has been the New York and Liverpool test for actual alkali, as per column No. 4 of table.

11. Analysis of Caustic Soda.—Dissolve 5 grams of the sample in distilled water in a 250-cubic centimeter measuring flask and make up to 250 cubic centimeters. This operation should be carried out without unnecessary exposure to the air as the caustic readily takes up water and carbon dioxide.

Determination of Sodium Oxide (Total Alkali).—Take 50 cubic centimeters of the above sample solution in a 200-cubic centimeter beaker and titrate cold with normal sulphuric acid, using 2 drops of methyl-orange indicator. In this titration, as the end point is approached, care should be taken to stir well, so that the color change from yellow to pink will be sharp and easily noticeable, the acid being added a drop at a time.

Cubic centimeters $n/H_2SO_4 \times 3.1$ = the per cent. of Na_2O
(total alkali) actual

Cubic centimeters $n/H_2SO_4 \times 3.2$ = the per cent. of Na_2O
(total alkali) New York and Liverpool test

Determination of Sodium Hydroxide.—Take 50 cubic centimeters of the above sample solution in a 200-cubic-centimeter beaker and add 100 cubic centimeters of barium-chloride solution, stir well, add 5 drops of phenolphthalein indicator, and titrate with normal sulphuric acid.

Cubic centimeters $n/H_2SO_4 \times 4$ = the per cent. of $NaOH$

Determination of Sodium Chloride.—Take 25 cubic centimeters of the above sample solution in a 200-cubic-centimeter

beaker, and add 5 cubic centimeters of colorless nitric acid of 1.42 specific gravity and about 2 cubic centimeters of iron-

TABLE I
COMPARISONS OF DIFFERENT SYSTEMS OF ALKALIMETRY
FOR SODA ASH

No. 1	No. 2	No. 3	No. 4
Soda Ash Sodium Carbonate Na_2CO_3 Per Cent.	Actual Alkali Sodium Oxide Na_2O Per Cent.	Newcastle Test Sodium Oxide Na_2O Per Cent.	N. Y. and Liv. Sodium Oxide Na_2O • Per Cent.
79.51	46.5	47.11	48.00
80.37	47.0	47.62	48.51
81.22	47.5	48.12	49.03
82.07	48.0	48.63	49.54
82.93	48.5	49.14	50.06
83.78	49.0	49.64	50.58
84.64	49.5	50.15	51.09
85.48	50.0	50.66	51.61
86.34	50.5	51.16	52.12
87.19	51.0	51.67	52.64
88.05	51.5	52.18	53.16
88.90	52.0	52.68	53.67
89.76	52.5	53.19	54.19
90.61	53.0	53.70	54.70
91.47	53.5	54.20	55.22
92.32	54.0	54.71	55.74
93.18	54.5	55.22	56.25
94.03	55.0	55.72	56.77
94.89	55.5	56.23	57.29
95.74	56.0	56.74	57.80
96.60	56.5	57.24	58.32
97.45	57.0	57.75	58.83
98.31	57.5	58.26	59.35
99.16	58.0	58.76	59.87
100.00	58.5	59.27	60.38

indicator solution. Run in a few drops of $n/20$ potassium-sulphocyanide solution from a burette, noting the quantity. Then titrate with $n/20$ silver nitrate, adding the silver solution

drop by drop and stirring constantly to decoloration, filter off the silver chloride formed, washing well on the filter with distilled water. Titrate the combined filtrate and washings to a permanent pink color with $n/20$ potassium sulphocyanide.

(Cubic centimeters $n/20$ $AgNO_3$ - Cubic centimeters $n/20$ $KCNS$) $\times .585$ = the per cent. of $NaCl$

Determination of Sodium Sulphate.—Take 50 cubic centimeters of the above sample solution in a 200-cubic-centimeter beaker, add 100 cubic centimeters of barium-chloride solution from a pipette drop by drop, and boil. To the boiling solution, add 10 cubic centimeters of distilled water and 5 cubic centimeters of concentrated hydrochloric acid. Boil for 10 minutes, and allow it to settle for 1 hour. Filter through an ashless filter paper, and wash the barium-sulphate precipitate thoroughly with hot water until the washings show no turbidity on adding silver-nitrate solution. Place the filter paper containing the barium-sulphate precipitate in a weighed platinum crucible and burn off the filter paper over a Bunsen burner at as low a temperature as possible. Finally, ignite to a full red heat until the barium sulphate is a good, white color. Cool and weigh. Weight of $BaSO_4$ in grams $\times 60.86$ = the per cent. of Na_2SO_4 .

Caustic soda is sold on its strength in Na_2O , as indicated in the New York and Liverpool test column in Table II.

The price is always based on 60-per-cent. caustic, with a proportionate addition for the higher percentages.

12. Analysis of Causticizing Lime.—The lime used in causticizing should be regularly tested in order to see that its quality is kept up to a reasonable standard. A chemical analysis will not necessarily show what results it will give in practice, but a simple causticizing test made under conditions similar to those of actual work, will give very valuable information. From each car of lime received, as fair a sample as possible should be taken by going all over the car and taking portions from the top, middle, and bottom of the load. This should be selected to represent both the fine and the coarse material, and as soon as the entire sample is taken it should be crushed and quartered down as rapidly as possible to avoid

TABLE II
COMPARISON OF DIFFERENT SYSTEMS OF ALKALIMETRY
FOR CAUSTIC SODA

No. 1	No. 2	No. 3	No. 4
Caustic Soda Sodium Hydroxide <i>NaOH</i> Per Cent.	Actual Alkali Sodium Oxide <i>Na₂O</i> Per Cent.	Newcastle Test Sodium Oxide <i>Na₂O</i> Per Cent.	N. Y. and Liv. Sodium Oxide <i>Na₂O</i> Per Cent.
74.83	58.0	58.76	59.87
75.48	58.5	59.27	60.38
76.12	59.0	59.77	60.90
76.77	59.5	60.28	61.42
77.40	60.0	60.79	61.93
78.05	60.5	61.30	62.45
78.70	61.0	61.80	62.97
79.35	61.5	62.31	63.48
80.00	62.0	62.82	64.00
80.65	62.5	63.32	64.52
81.29	63.0	63.83	65.03
81.94	63.5	64.33	65.55
82.58	64.0	64.84	66.06
83.23	64.5	65.35	66.58
83.87	65.0	65.85	67.10
84.52	65.5	66.36	67.61
85.16	66.0	66.87	68.13
85.81	66.5	67.37	68.65
86.45	67.0	67.88	69.16
87.10	67.5	68.39	69.68
87.74	68.0	68.89	70.19
88.39	68.5	69.40	70.71
89.03	69.0	69.91	71.23
89.67	69.5	70.41	71.74
90.30	70.0	70.92	72.26
90.95	70.5	71.43	72.77
91.60	71.0	71.93	73.29
92.25	71.5	72.44	73.81
92.90	72.0	72.95	74.32
93.55	72.5	73.45	74.84
94.19	73.0	73.96	75.35
94.84	73.5	74.47	75.87
95.48	74.0	74.97	76.39
96.13	74.5	75.48	76.90
96.77	75.0	75.99	77.42
97.32	75.5	76.49	77.94
98.06	76.0	77.00	78.45
98.71	76.5	77.51	78.97
99.35	77.0	78.01	79.49
100.00	77.5	78.52	80.00

the absorption of moisture. From this final sample a weighed amount should be taken and boiled for exactly 1 hour with water and a weighed amount of dry soda ash which is in excess of the amount the lime can causticize. The amount of water used should be so taken that the final solution at the end of the test is about the strength of that used in practice. After the boiling is completed, the sludge should be allowed to settle and the clear liquor titrated with both phenolphthalein and methyl orange as in the case of cooking liquor. Knowing the causticity from this titration and the weight of soda ash taken, the amount of lime required to causticize 100 lb. of dry soda ash may be calculated by this formula:

$$\frac{\text{Weight of lime used} \times 100}{\text{Per cent. of causticity} \times \text{weight of soda ash used}}$$

The settling quality of the lime may be ascertained in this same test by taking a sample of the rapidly boiling mixture just at the end of the test and, without giving it time to settle, filling a 100-cubic-centimeter graduate just to the upper mark. By noting the cubic centimeters of clear liquor at fixed time intervals, the relative settling qualities of the various limes can be compared.

13. Analysis of Sludge from Causticizing Pans.—The lime mud which settles in the causticizing and washing tanks is generally a waste product, and in order to see that too much caustic soda is not thrown away, it should be tested at intervals for the amount of alkali present. A representative sample of the dried mud is weighed out, placed in a small porcelain dish, and moistened with a strong ammonium-carbonate solution. It is next evaporated to dryness, then heated over a low flame until no odor of ammonia can be noticed, and finally it is leached out repeatedly with boiling distilled water until all the soluble alkali is removed. The combined leachings are then titrated with standard acid, using methyl orange as indicator, and the results calculated to percentage of alkali based on the dry lime mud.

14. Analysis of Bleaching Powder.—The value of bleaching powder depends on the percentage of available chlorine in it.

On standing, bleaching powder loses its strength, especially if kept in a damp place. A good bleaching powder should contain from 36 to 37 per cent. of available chlorine, but it is generally accepted if it tests over 35 per cent.

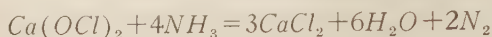
Sampling.—Samples should be taken immediately upon arrival of shipment from 10 per cent. of the casks. The sampling may be carried on by driving a $\frac{3}{4}$ -inch brass tube through the cask from end to end, and transferring the powder inside to a quart Mason jar. This should be sealed and taken to the laboratory, where the composite sample is to be rapidly quartered down to several ounces, and the lumps smashed up finely.

Available Chlorine.—About 5 grams should be weighed out from the weighing bottle and triturated in a mortar with several portions of water, each portion being transferred to a 1,000 cubic-centimeter graduated flask. Finally, the paste, after being thoroughly ground, is washed into the flask and the solution made up to 1,000 cubic centimeters. Shake well and draw off with a pipette 50 cubic centimeters of the solution. Transfer to a beaker and add 1.5 grams of potassium iodide freshly dissolved, dilute with 50 cubic centimeters of water and acidify with acetic acid. Titrate with $n/10$ sodium-thiosulphate solution and calculate the per cent. of available chlorine.

Quality of Powder for Producing Bleach Liquor.—Sixty grams of bleaching powder are weighed out and added to 1,000 cubic centimeters of distilled water in a glass jar $4\frac{1}{2}$ inches in diameter, and 5 inches deep. The mixture is stirred vigorously by means of a glass rod bent so that it just clears the bottom and side of the jar, driven by power from an electric or water motor at 250 revolutions per minute for exactly 15 minutes. The jar is then placed on a bench which is free from vibration, and the time required for the dregs to settle to the bottom is noted. Of the clear bleach liquor, 25 cubic centimeters is measured out and tested for available chlorine in the usual way.

The foregoing determinations will usually be found sufficient, but if it is desired to test for chlorides and chlorates, the determinations may be carried out as follows: Measure 25

cubic centimeters of solution into a beaker, add 10 cubic centimeters of dilute ammonia, cover the beaker and boil the solution gently for 5 minutes. The hypochlorite is then converted into chloride, according to the following equation:



Add in excess dilute nitric acid, carefully avoiding loss through effervescence, and boil until free from carbon dioxide. Then add an excess of $n/10$ silver-nitrate solution and stir until the precipitate coagulates. Filter, wash, and titrate the excess of silver nitrate in the filtrate with standard potassium sulphocyanide. From the total chloride thus found, subtract the hypochlorite.

Hypochlorite, Chloride, and Chlorite.—Boil 25 cubic centimeters of solution with dilute ammonia and pass SO_2 through the boiling solution for a few minutes, in order to reduce the chlorate to chloride. Acidify with dilute sulphuric acid and oxidize the excess of sulphur dioxide by carefully adding potassium permanganate until the solution is faintly pink. All the chlorine in the bleaching powder is now present in the solution, as chloride. Determine by means of standard silver nitrate and potassium sulphocyanide solutions and calculate to chlorate.

15. Analysis of Bleach Sludge.—It is important that the bleach sludge should be tested from time to time to ascertain the loss of available chlorine in the dump. The sludge, after drawing off the wash as closely as possible, should be agitated and samples taken.

Determination of Available Chlorine.—Measure 50 cubic centimeters of the well-mixed sample into a beaker by means of a pipette, which is rinsed out into the beaker. Titrate with $n/10$ sodium-arsenite solution as before. The number of cubic centimeters used multiplied by .071 gives the number of grams of available chlorine per liter of sludge, and this multiplied by 3.785 (the number of liters in 1 gallon) gives the weight of available chlorine in a gallon. This weight multiplied by three gives the approximate loss of bleaching powder in a gallon, and, knowing the cubical contents of the tank and the number

of inches dumped, the loss in the dump can be easily calculated. In a bleach that settles well, this loss ranges from .6 per cent. to 1 per cent. of the bleach mixed. If the bleach does not settle well, the loss will reach 3 per cent. and may go even higher.

16. Analysis of Black Ash.—Black ash is the ash that is recovered after the liquor used in the soda process, in which the wood has been cooked, has been evaporated and the residue burned.

Preparation of Sample.—Quite a large sample should be taken, ground up, mixed, and quartered several times. The last portion should be ground very fine and well mixed in the sample bottle. Weigh out 50 grams of this carefully prepared sample and boil up with water. Stir well from time to time for at least an hour, filter into a liter flask, wash well, cool, and dilute to the mark. With black-ash waste, the volume of carbon is relatively so large that it is well to burn off most of it in a platinum dish before attempting to leach out the soda. In the analysis of black ash it is customary to determine only the caustic soda or soluble alkali present in the solution obtained by leaching.

Determination of Caustic Soda.—Take 50 cubic centimeters of the clear liquid and precipitate the carbonate with a slight excess of neutral barium-chloride solution in a 200-cubic-centimeter flask, add hot water, shake well, cool, fill to the mark, and after further shaking, allow it to settle. Take 100 cubic centimeters of the clear portion and titrate with normal acid, using methyl orange as an indicator. Multiplying the number of cubic centimeters of acid used by 4, and dividing by 1.25, will give the percentage of NaOH . This also includes any Na_2S present, and if it is considerable, the number of cubic centimeters of standard acid neutralized by it must be deducted from the whole before the calculation of the caustic soda is made.

17. Analysis of Alum.—In the paper industry, the term *alum* is applied to sulphate of aluminum $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$, which, strictly speaking, is not an alum. The analysis of an

alum is quite long and complicated, but, if carried on with care, very good results can be obtained.

Sampling.—If the shipment is received in packages, samples should be drawn from 5 per cent. of them, and these samples should be thoroughly ground and mixed into one composite sample representing the shipment.

If the shipment is in ingots, fragments chipped off from twelve ingots, taken at random, should be taken as representative samples, which should be ground together into one composite sample representing the shipment.

Determination of Water.—Weigh out 2 grams of the well-mixed sample into a platinum crucible. Heat gradually until a low red heat is reached, and allow it to remain at this temperature until fumes of SO_2 are perceptible. Cool in a desiccator and weigh. The loss of weight gives the amount of water, together with some SO_2 . The loss of SO_2 may be determined by adding about 10 cubic centimeters of hot, concentrated, hydrochloric acid to the contents of the crucible, allowing the solution to stand in a warm place for about $\frac{1}{2}$ hour, when the lumps will all be broken down, provided the heating was not too high or too low when driving off the water. The contents of the crucible should then be washed with hot water into a small beaker, diluted to about 50 cubic centimeters, and heated until all is dissolved, except the little insoluble matter there may be in the alum. Filter and wash well with hot water. Precipitate with sulphuric acid in the filtrate with barium chloride, as in the determination of sulphuric acid. Filter, wash well with hot water, dry, and ignite to constant weight. The weight of $BaSO_4$ multiplied by .34335 will give the weight of SO_3 . This same weight subtracted from the weight of the total SO_3 in the same weight of sample, as found by separate determination, will give the loss of weight due to SO_3 . Subtracting this from the total loss on heating will give the loss due to water, and this multiplied by 50 will give the percentage of water in the alum.

Insoluble Matter.—Weigh out 10 grams of sample in a beaker and dissolve in about 200 cubic centimeters of hot distilled water. Filter through a weighed Gooch, or alundum,

crucible, using suction, wash well with hot, distilled water, dry to constant weight, and calculate the per cent. of insoluble matter.

Alumina and Iron Oxide.—Make up the filtrate from the insoluble matter to 1 liter, measure out 100 cubic centimeters (equal to 1 gram of the original sample) and add 100 cubic centimeters of distilled water and 5 cubic centimeters of concentrated hydrochloric acid, and a few drops of concentrated nitric acid (1 cubic centimeter of a 5-per-cent. solution of tannin, if added at this time, will coagulate the gelatinous precipitate, and render it more easily filterable). Heat the solution to boiling and add dilute ammonia drop by drop with constant stirring until a slight excess is present, or until the solution is distinctly alkaline. Boil 1 minute, allow the precipitate to settle, and filter promptly. Wash by decantation, using hot water containing a small amount of ammonia and ammonium nitrate. Continue washing until the filtrate no longer shows the presence of chlorides when tested with silver-nitrate solution. Then wash precipitate through a hole in the filter paper back into beaker in which it was precipitated, and dissolve in 5 cubic centimeters of concentrated hydrochloric acid, add a few drops of concentrated nitric acid, make up the volume to about 300 cubic centimeters, and reprecipitate the aluminum and iron with ammonia as above. The double precipitation will eliminate any alkali earth which, if present, should be carried down with the first precipitation. The precipitate is partly dried and then ignited in a platinum crucible, and finally heated to a constant weight with a blast lamp, or a Meker burner. Thirty minutes is usually sufficient. Calculate the percentage of Al_2O_3 and Fe_2O_3 .

If basic sulphate of aluminum is present, it may be advisable to look for aluminum in the filtrate from the first precipitation. This may be done by adding a slight excess of hydrochloric acid. Evaporate to 200 cubic centimeters, make it alkaline with ammonia and continue the evaporation to 100 cubic centimeters, occasionally adding a drop of ammonia. The small amount of aluminum hydroxide, if present, is filtered off and added to the main portion.

Sulphate.—Barium-sulphate precipitation carries down appreciable amounts of aluminum when it is present in quantity. Hence the aluminum should be removed before precipitating the sulphate.

18. Dilute the filtrate from the iron and aluminum determination to about 300 cubic centimeters, neutralize with hydrochloric acid, and add exactly 1 cubic centimeter of concentrated hydrochloric acid. To the boiling solution, add an excess of a hot 5 per cent. solution of barium chloride. This must be added slowly, a few cubic centimeters at a time, with constant stirring. Allow the precipitate to settle on a hot water bath from 20 minutes to a half hour. Decant the liquid through a weighed Gooch crucible or ashless filter paper and wash the precipitate four times with 50 cubic centimeters of hot water. Transfer the precipitate to the filter and wash with hot water until the washings no longer give a test for barium with potassium chlorate. Dry and ignite the precipitate gently over a Bunsen burner for $\frac{1}{2}$ hour. (Do not heat over a blast lamp.) Any barium sulphide formed by combustion of filter paper, on being gently ignited in an inclined crucible, is changed back to barium sulphate. Calculate the weight of SO_3 .

Iron.—Dissolve 5 grams of alum sample in 100 cubic centimeters of hot water, adding 5 cubic centimeters of concentrated sulphuric acid, and heat the solution nearly to boiling. Add potassium-permanganate solution drop by drop to a permanent, strong, pink color to oxidize any possible reducing matter. Run the solution through a Jones reductor in the usual manner, cool, and titrate with standard potassium-permanganate solution.

If a Jones reductor is not available, the ferric salt may be reduced with hydrogen sulphide. The solution is placed in a 200-cubic-centimeter flask, filtered with a stopper provided with two tubes for gas to enter and leave the flask. The contents of the flask are heated to boiling and hydrogen sulphide passed through the solution until it is perfectly colorless. The boiling is continued and carbon dioxide is now passed through the solution until the excess of hydrogen sulphide is entirely

removed. Cool and titrate with standard permanganate as above. Calculate the percentage of Fe_2O_3 .

Determination of Free Acid.—It is very important in determining the value of an alum to know the amount of free acid present, as this attacks the coloring matter and at the same time injures the machinery of the mill. Many methods have been suggested for the quick determination of the free acid, but they are all very satisfactory. Three methods are here given:

1. Digest a weighed quantity of alum for about 15 hours in strong alcohol. Filter, wash with alcohol of the same strength and titrate with $n/10$ alkali, using phenolphthalein as an indicator. Calculate the percentage of free acid.

2. Dissolve 2 grams of alum in 5 cubic centimeters of water and add 5 cubic centimeters of cold, saturated, solution of ammonium sulphate, stirring thoroughly. Now add 50 cubic centimeters of 95-per-cent. alcohol, filter, evaporate the filtrate on a water bath, take up in water and titrate with $n/10$ alkali, using phenolphthalein as an indicator.

3. The following is the most accurate method for the determination of the free acid in alum: First make a complete analysis of the alum. Determine the amount of sulphuric acid necessary to combine with all the bases. If there is more acid than is necessary to combine with all the bases, the excess may be considered as free sulphuric acid. If there is not acid enough to combine with all the bases, combine the acid with all the bases except alumina, and then use up all the latter possible, reporting the balance as basic alumina Al_2O_3 .

Sizing Test.—The standard size is prepared as previously described. Dissolve 10 grams of alum in water and dilute to 1 liter. Fill a 200-cubic-centimeter flask about two-thirds full with distilled water, and run in from a burette 20 cubic centimeters of size solution. Now add the alum solution from a burette, a few drops at a time. Shake violently, and allow the precipitate to rise after each addition. Toward the end the alum should be added a drop at a time. When the precipitate on rising leaves the solution perfectly clear, take the reading of the alum burette. The number of cubic centimeters of alum solution multiplied by .01 will give the number of grams of

alum necessary to precipitate 20 cubic centimeters of standard size and will give the number of parts of neutral dry size precipitated by 1 part of alum.

19. The foregoing is a good quick test for the precipitating power of alum, but it does not give much of an idea as to the quality of alum, as it may be a very acid alum and still have a good precipitating power. Table III gives analysis of alums.

In sample 1 the high percentage of water insoluble material indicates that the raw material was not entirely dissolved by

TABLE III

ANALYSES OF ALUMS

From "Modern Pulp and Paper Making," by G. S. Witham

	1	2	3	4
Insoluble in water.....	10.61	.67	.11	.02
Alumina (Al_2O_3).....	14.96	22.37	11.64	22.02
Iron oxide (FeO).....	.13	.46	.06	none
Iron oxide (Fe_2O_3).....	1.08	.08	1.17	.01
Zinc oxide (ZnO).....	none	3.80	none	none
Soda (Na_2O).....	.57	none	4.75	.72
Magnesia (MgO).....	none	none	.45	none
Combined sulphuric acid.....	37.36	45.28	35.98	45.36
Free sulphuric acid.....	1.08	none	5.13	none
Water by difference.....	34.21	27.34	40.71	31.87
Sizing test (parts of neutral rosin size precipitated by one part of the alum)	3.47	3.64	3.19

the acid. The free acid and the iron are also too high in this sample. Such an alum is more suitable for water softening than for paper making. Sample 2 is a better alum but the iron is too high. Sample 3 is a very poor alum, the alumina being very low and the percentage of free acid being abnormally high. Sample 4 is one of the best commercial alums on the market today. The alumina is high, the iron almost negligible and no trace of free acid present. It is interesting to note that

apparently a higher alumina content than that required for the neutral sulphate has no effect on the sizing efficiency of the alum, as sample 2 in which the alumina is very high is not appreciably more efficient than sample 3 in which the alumina is very low.

FILLERS

20. Testing of Fillers.—Fillers such as clay, talc, agalite, asbestine, ground filler, etc., are not usually subject to chemical analysis. The two most important factors to determine about these substances are: (*a*) color; and (*b*) the amount of grit present.

The usual method of determining the color of fillers is by comparing the sample with a standard sample. Such comparisons should always be made under equal conditions of dryness, since the amount of moisture present has a marked influence on the color. The samples should be dried 100° C. for some time before comparing them.

The amount and kind of grit can be determined by washing tests, as described in connection with the testing of clay, followed by microscopical examination. Photographic reproductions of the appearance of the various fillers under a microscope can be obtained from the United States Bureau of Standards, and the paper-mill chemist can soon familiarize himself with the appearance of desirable and undesirable fillers under the microscope. This is a matter in connection with which it is impossible to give detailed written instructions; because practice and experience are necessary.

As clay is considerably more important to the paper maker than any other filler material, the following tests for clay, which are those in use in the Arthur D. Little Laboratory, are described in some detail.

21. Testing of Clay.—Clay, or kaolin, for use as a filler in high-grade papers should be pure, white, and free from artificial bluing. It should not have an appreciably gritty feel when rubbed between the teeth. The best clays will not show more than 1 to 2 per cent. residue by the flotation test, nor

more than a few tenths of 1 per cent. (generally less than .25 per cent.) by the 200-mesh sieve test. They should not contain an excessive amount of free moisture when shipped.

Moisture.—Dry 10 grams to constant weight at 100 to 105° C. The loss in weight is considered as moisture.

Grit.—1. Flotation Test: Measure a depth of 2 inches from the bottom of a 500-cubic-centimeter beaker and make a mark on the beaker to indicate this height. Weigh 20 grams of the clay into this beaker, mix thoroughly with water, and fill up to the mark. Let it settle for exactly 1 minute and pour off the milky water. Repeat the process until the supernatant water can be poured off practically clear at the end of a minute. Place the beaker on the steam bath until perfectly dry, brush out the settled grit into a balanced watch glass with a camel's-hair brush, and weigh it.

2. 200-Mesh Sieve Test: Place 10 grams of clay in a 200-mesh sieve and wash by means of a slow stream of running water and finally with a wash bottle until all the clay has been washed through the sieve. The residue which does not pass through is then washed into a small beaker and, after evaporating off the water on the steam bath, it is brushed into a balanced watch glass and weighed.

Color.—Spread out a small amount of clay (preferably dried at 100°C.) on white paper by the side of, and in contact with, a similar amount of a standard sample. Each should be pressed down with a spatula to give a smooth surface. The color is then compared. When the clays are of the same color, it is impossible to see any line of demarcation between them. If a permanent record is desired, the color should be measured by a colorimeter, or tintometer.

Artificial Coloring.—In one of two similar white porcelain dishes place a measured amount of freshly saturated lime water, and in the other dish an equal amount of clear, distilled water. Then into each of these liquids, dust, from the end of a knife or spatula, a little at a time, equal amounts of clay. If the clay has been artificially blued, the lime water will remove the bluing. After letting it stand for a few moments, the excess liquid should be siphoned off and the moist clay

examined. If the clay has been blued, the residue from the lime-water treatment will show the original color, which will be quite different from the color shown by the moist clay treated with water alone in the other dish.

22. Retention of Clay and Other Fillers.—The proportion of the filler added to the engine which appears in the finished paper is spoken of as the retention. This may vary from 30 to 90 per cent., though the latter figure is only reached under

TABLE IV
FILLER RETENTION

Filler	Percentage in Finished Paper	Percentage Retention
China clay.....	39.1	76.2
China clay.....	32.2	74.6
China clay.....	26.7	68.6
China clay.....	25.6	66.5
China clay.....	15.1	57.6
Precipitated chalk.....	24.8	38.7
Precipitated chalk.....	19.1	40.9
Precipitated chalk.....	12.0	36.6
Precipitated chalk.....	9.7	54.0
Asbestine	22.3	72.4
Asbestine	10.8	46.1
Asbestine	10.2	52.4
Pearl finish.....	24.9	64.4
Crown filler.....	24.2	51.2
Blanc fixe.....	27.9	39.0

very exceptional circumstances and a retention of 50 per cent. is generally considered satisfactory. Many factors, other than the filler, influence its retention, and, except in a general way, it is impossible to estimate their effects. The kind of stock and the extent of its beating, the speed of the paper machine, the pull on the suction boxes, the amount of filler added and the thickness of the sheet all must be taken into consideration. Slow, or *greasy*, stock, a light suction, and a thick sheet all tend to give high retention, while in the case of sulphate of calcium the retention increases with the amount added.

Working with sulphite, and with sulphite and soda furnishers, the retention has been found to decrease slightly as the amount of clay added was increased. This is contradictory to the results given from observations. It has also been found, as would be expected, that increasing the thickness of the sheet—the ream weight—increased the retention. It was also increased as the amounts of size and alum were increased, and by greater hydration of the stock in the beater.

Table IV gives results of tests made by Sutermeister, as to what may be expected from various fillers when used in manufacturing high-grade book papers.

23. Analysis of Rosin.—Rosin should be tested for acid number, or saponifiable matter, unsaponifiable matter, percentage insoluble in petroleum ether, and practical sizing tests.

Determination of Acid Number, or Amount of Caustic Potash Required to Neutralize One Gram Rosin.—Acid number is best determined by dissolving a weighed sample of the rosin in neutral alcohol and titrating directly with alcoholic KOH solution, using phenolphthalein as indicator. With average American rosin about 164 milligrams of KOH will be required for every gram of rosin. This test indicates roughly the amount of alkali which the rosin will use up in the ordinary size-making process, and it may be used as the basis for calculating the reduction in the amount of alkali which should give a size with a definite percentage of free rosin.

Determination of Unsaponifiable Matter.—The unsaponifiable matter may be determined by heating a sample on the steam bath for several hours with an excess of caustic-potash solution, cooling, extracting with ether, as in the case of size, evaporating off the ether, drying, and weighing. This unsaponifiable matter is soft and sticky in character, and, if present in large amount, is likely to cause trouble by sticking at various points in the paper machine.

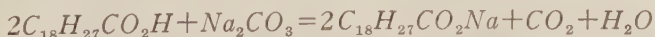
Determination of Insoluble Matter.—The amount of material insoluble in petroleum ether should be determined by dissolving a weighed sample of the rosin in petroleum ether, separating

the solution from the insoluble residue, washing the latter with several portions of ether, and then drying and weighing. The insoluble matter has practically no sizing properties, and it should be present in a small amount only.

Practical Sizing Tests.—For practical sizing tests, a small beating engine is desirable, although the work can be done by using a cream whipper. Twenty-five or 50 grams (dry weight) of unbleached, sulphite pulp is thoroughly disintegrated in the beater, or cream whipper, and 2 per cent. of rosin sizing added in the form of a thin milk. After thoroughly mixing, 3 per cent. of a standard alum is added in solution and thoroughly mixed. The pulp is then thinned, made into hand sheets and the dried sheets are tested for ink penetration. Two sets of sheets are made, one with size made from a standard rosin and the other with size made from the rosin under test. The size is made by cooking a given weight of powdered rosin, in a container surrounded by boiling water, for 4 hours with that weight of soda ash, sodium carbonate, which will yield a size containing 25 per cent. free rosin only on the dry basis, that is, with sufficient sodium carbonate to neutralize 75 per cent. of the free acid in the rosin. The water used in making up the size should be sufficient to give a finished thick size containing 30 per cent. dry matter. The thick size is diluted to a milk by stirring with water at 70° F. before adding the pulp.

ANALYSIS OF SIZE OR GLUE

24. Analysis of Rosin Size.—When rosin and soda are boiled together, they combine to form resinate of soda, or rosin soap. The abietic acid and the sodium carbonate are supposed to react according to the following equation:



Rosin size, prepared by different processes, is analyzed by the same methods.

Free Rosin.—Weigh out approximately 10 grams of size and mix with 30 cubic centimeters of water. With solid or dry size 6 to 8 grams will be sufficient. Wash with as little

water as possible into a 500-cubic-centimeter separatory funnel, and free it from any trace of acid, or alkali. Extract with 25 cubic centimeters of acid-free ether. Draw off the watery layer into a second separatory funnel, extract this with a second portion of acid-free ether, and add the ether extract to that in the first funnel. Wash the combined ether extracts with two 25-cubic-centimeter portions of water, adding the wash waters to the solution in the other funnel. Pour the washed ether extract into a weighed Soxhlet flask. Finally, extract the water solution a third time with 25 cubic centimeters of ether, first using the ether to rinse out the funnel which contained the ether extracts. Draw off the watery layer into another separatory funnel, wash the third ether extract twice with 25 cubic centimeters of water. Draw off the water each time into the funnel containing the soap solution. Pour the washed ether extract into the Soxhlet flask containing the main ether extract. Distill off the ether and dry the flask at not over 105°C. to constant weight. Cool in a dessicator and weigh the free rosin.

Moisture.—Run the residue from the free-rosin determination into a 350-cubic-centimeter flask, or a 500-cubic-centimeter flask, if necessary. Dilute to the mark and mix thoroughly. Pipette an aliquot equivalent to one-tenth of this solution into a weigh platinum dish, evaporate to dryness on the water bath, place in a water oven, and dry at 105°C. to constant weight. Two hours' drying ought to be sufficient. Divide this weight by one-tenth of the weight of the sample taken and multiply by 100. This gives the per cent. of dry matter in the size, exclusive of free rosin. Add the per cent. of free rosin, as above determined, and subtract the sum from 100; the difference will be the per cent. of water.

Total Alkali.—Ignite the residue from the moisture determination until all carbonaceous matter is burned off. Cool in a desiccator and weigh. Dissolve the residue in a few cubic centimeters of water and titrate with $n/10$ acid and methyl orange. Calculate the titration directly to sodium carbonate. This weight should check the weight of ash reasonably close, unless the size contains insoluble, or other foreign, matter.

Calculate the titration also to sodium oxide; divide the weight thus obtained by one-tenth of the original sample taken, and multiply by 100. This gives the per cent. of sodium oxide in the size.

Calculation: 1 cubic centimeter $n/10$ acid = .0031 gram Na_2O = .0053 gram Na_2CO_3 .

Combined Rosin.—Pipette an aliquot representing four-fifths of the soap solution from the determination of free rosin into a separatory funnel and acidify with 10 cubic centimeters of dilute sulphuric acid (1:5). Add 25 cubic centimeters of ether, shake well, and let stand until the two layers are completely separated. Draw off the water solution into the second separatory funnel and wash the ether with two 25-cubic centimeter portions of water, drawing off the water into the second funnel and pouring the ether extract into a weighed Soxhlet flask. Rinse the first funnel with 25 cubic centimeters of ether into the second funnel. Shake well and draw off the water layer into the first funnel. Wash as already described with two 25-cubic-centimeters portions of water. Repeat once more. Evaporate the ether from the combined extracts as in the free-rosin determination. Dry to constant weight at not over $105^{\circ}C$. Divide the weight obtained by .8 of the original weight of the sample taken and multiply by 100 to obtain the per cent. of combined rosin.

Free Carbonate.—Weigh out 10 grams of size and dissolve in 200 cubic centimeters of acid-free absolute alcohol. Let the solution stand 8 to 10 hours, or overnight if possible, protected from acid fumes and moisture. Filter on a weighed dry filter and wash thoroughly with absolute alcohol. Pour boiling water through filter, and after cooling, titrate the aqueous solution with $n/10$ acid and methyl orange. Calculate to sodium carbonate.

Calculation: 1 cubic centimeter $n/10$ acid = .0053 gram Na_2CO_3 .

25. Testing of Glue.—Glue, or animal size, is made by heating the clippings of bones, horns, etc., with water. The best grades of glue for paper-mill use have been thoroughly

washed, and are ready for mixing up for sizing purposes without any previous purification. Glue is insoluble in cold water, but will absorb six to eight times its weight of water, and swell up. The best glue should absorb eight times its weight of water at a temperature of 15° C. in 24 hours. Glue is readily soluble in hot water, the solution gelatinizing on cooling. The following tests may be made to determine the quality of glue for paper makers' use:

Determination of Power of Absorption.—Weigh out 10 grams of the sample into a beaker and fill with cold water, taking care that all of the glue is covered by the water. Do not allow the temperature to rise above 15° C. during 24 hours. At the end of that time, pour off the excess of water and weigh again. The increase of weight will indicate the amount of water absorbed. The absorptive power is expressed as the number of times its weight of water absorbed.

Moisture.—Weigh 2 or 3 grams of the glue into a platinum dish, add 10 to 15 cubic centimeters of hot water, mix thoroughly with a small stirring rod and rinse the rod off into the dish with a wash bottle. Evaporate on the steam bath to dryness and then dry to constant weight at 105°C. The loss in weight of the original glue represents the moisture. A good glue contains not less than 8 per cent. and not more than 16 per cent. of moisture. Low moisture indicates overheating in the manufacture.

Ash.—Ignite the residue from the moisture determination in a platinum dish. Use a low heat at first, for the reason that too rapid burning will make it very difficult to get rid of the last of the carbon. Then raise the heat and ignite to constant weight at the full heat of a Bunsen or Tirrill burner. Report the residue as ash.

In making the ash determination, note whether the ash has fused or not. The ash of bone glue fuses and the aqueous solution is neutral, containing phosphates and chlorides. The ash of hide glues does not fuse, owing to traces of lime which render the aqueous solution slightly alkaline. This solution is also free from phosphates or chlorides. The ash will vary from 2 to 8 per cent. according to the quality of the glue.

The ash may also show the presence of alum, which is sometimes added to impart a fictitious *body* to glues.

Grease.—Make a strong water solution of aniline blue. Use 25 per cent. solutions of the different samples of glue to be tested. To 25 cubic centimeters of the glue solution add 2 cubic centimeters of the dye, and with a 1.5-inch flat, camel's-hair brush, draw a smooth streak of the dyed glue across wrapping paper. White spots indicate grease. A comparison of the white spots will indicate the relative amount of grease in the different samples.

Grease may be determined quantitatively by extracting 5 to 10 grams of the ground and dried material with anhydrous ether in a Soxhlet extractor, evaporating the ether and weighing the residue.

Comparison of the Value of Glues.—A good practical test that serves as a comparison of two glues as to money value is made as follows: Weigh out 10 grams of the higher-priced glue in a small beaker, and then weigh out an amount of the lower-priced glue that will be equivalent in price to the 10 grams of the former. Add 100 cubic centimeters of water to each at the same time and heat gently, keeping both at the same temperature and stirring in the same manner until all the glue is dissolved. Remove and allow to cool and gelatinize. If neither gelatinizes, it will be necessary to take larger samples. A good glue for paper makers' use should gelatinize at 75°F. when mixed in the proportion of 10 grams of glue to 100 cubic centimeters of water.

Illustration: Two glues marked A and H, and costing 12½ cents and 12 cents, respectively, were compared. Treated in the manner just described, it required 11.5 grams of H to gelatinize from the same amount of water as 10 grams of A. Therefore, if H is worth 12 cents per pound, A is worth 13.8 cents, and as A sells at 12½ cents a pound, other things being equal, it is by far the cheaper glue.

SULPHUR ANALYSIS

26. Sulphur is used in the manufacture of acid, or bisulphite liquor, for making sulphite pulp. For every 100 kilograms of sulphur, there should be a yield of 190 kilograms of sulphur dioxide. A good sulphur should contain less than 1 per cent. of foreign matter.

Moisture.—Grind the sample and weigh out 2 to 5 grams in a flat-bottomed dish. Dry to constant weight at a temperature not exceeding 105°C . Report the loss as moisture.

Sulphur.—Dissolve the residue from the moisture determination in CS_2 . Filter through a weighed Gooch crucible, which has previously been treated with a little CS_2 . Wash the residue with CS_2 and then with a little ether. Dry and weigh. Subtract the percentage insoluble in CS_2 from 100 per cent.

Ash.—Weigh out 2 to 5 grams in a porcelain crucible; ignite gently at first and finally in a full Bunsen flame. Cool the ash in a desiccator and weigh.

GAS ANALYSIS

27. Analysis of Sulphur Burner Gases.—In order to control the burners properly, the gases should be tested once an hour, or oftener if the burners are not working satisfactorily. The sulphur dioxide may be determined by means of an Orsat apparatus, Fig. 2, using caustic soda in the absorption pipette. The gas is measured in the burette over water, which soon becomes saturated with SO_2 , and then introduces only a very slight error.

Excess air may be estimated by determining the oxygen by means of alkaline pyrogallate after first absorbing the SO_2 in caustic soda. This test gives valuable information regarding leaks between the combustion chamber and the absorption system.

Sulphur trioxide in the gases is very difficult to determine with accuracy. It can be done by passing the gas at a rate of 1,000 cubic centimeters in 20 to 25 minutes, first through a hard glass sampling tube, surrounded with an iron jacket, and then through a tube 30 centimeters long, which is filled with

garnets and bits of porcelain, and cooled with ice. The gas is measured by the amount of water delivered by the syphon bottle which is used to induce its flow. After passing 2 to 5 liters of gas, the tube is washed out by drawing pure air through it and finally is washed into a beaker with water to remove the SO_2 . This is then determined gravimetrically by the precipitation as barium sulphate. The generally accepted place to sample the gases is in the main pipe between the cooler and the absorption apparatus.

Besides these chemical tests, it is customary to record the temperature of the gases as they leave the combustion chamber, and again after passing the cooler. Some form of recording pyrometer is desirable for the former work while an ordinary chemical thermometer is satisfactory for the latter.

28. Flue - Gas Analysis.—The most ordinary use of the Orsat apparatus is for the analysis of flue gases from power

plants in order to determine the efficiency of combustion. However, it can be used for the analysis of any kind of mixture of gases; for instance, burner gases from pulp-mill sulphur burners. Naturally, the solutions used in the pipettes will vary in accuracy with the gas being analyzed.

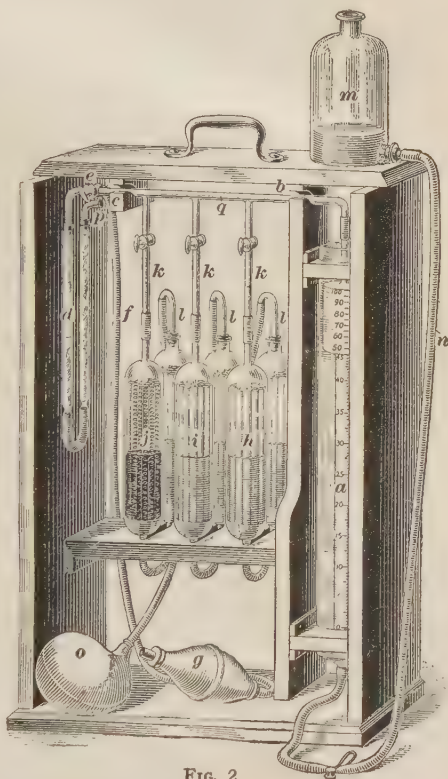


FIG. 2

The burette *a*, Fig. 2, is graduated in cubic centimeters up to 100, and is surrounded by a water jacket to prevent any change in temperature from affecting the volume of gas during analysis. For accurate work on flue gases, it is advisable to use three pipettes, *h*, *i*, and *j*, the first containing a solution of caustic soda for absorbing carbon dioxide, the second an alkaline solution of pyrogallol for absorbing oxygen, and the third an acid solution of cuprous chloride for absorbing carbon monoxide. Each pipette contains a number of glass tubes to which some of the solution clings, thus facilitating absorption. In pipette *j*, these tubes contain copper wire to strengthen the cuprous chloride solution as its absorbent power becomes weakened. The rear half of each pipette is closed by a rubber stopper fitted with a small glass tube which is connected with the rubber bag *o*, to protect the solution from the action of the air. The solution in each pipette should be drawn up to the mark on the capillary tube. Block *c* holds the apparatus in place.

The gas is drawn into the burette through the U-tube *d*, which is connected to the flue or to the sampler at *p*, and is filled with spun glass, or similar material, to clean the gas. To discharge any air or gas in the apparatus, the cock *e* is opened to the air and the bottle *m* is raised until the water in the burette reaches the 100-cubic-centimeter mark. The cock *e* is then turned so as to close the air opening and to allow gas to be drawn through *q*, the bottle *m* being lowered for this purpose. The gas is drawn into the burette to a point below the zero mark, the cock *e* then being opened to the air and the excess gas expelled until the level of the water in *m* and in *a* are at the zero mark. This procedure is necessary in order to obtain the zero reading at atmospheric pressure. The tube *n* connects bottle *m* with burette *a*.

29. The apparatus as well as all connections leading thereto should be carefully tested for leakage. Simple tests can be made. For example, if, after the cock *e*, Fig. 2, is closed, the bottle *m* is placed on the top of the frame for a short time and again brought to the zero mark, the level of the

water in *a* is above zero mark, a leak is indicated. Before taking a final sample for analysis, the burette *a* should be filled with gas and emptied once of twice, to make sure that all the apparatus is filled with new gas. The cock *e* is then closed, the cock *k* opened, and the gas driven over into *h* by raising *m*. The gas is drawn back into *a* by lowering *m*, and when the solution in *h* has reached the mark in the capillary tube, the cock *k* is closed and a reading is taken on the burette, the level of the water in the bottle *m* being brought to the same level as the water in *a*. The operation is repeated until a constant reading is obtained, the decrease in volume, in cubic centimeters being the percentage of the carbon dioxide (CO_2) in the flue gases. The gas is then driven over into the pipette *i*, and the apparatus manipulated in a similar manner. The difference between the resulting reading and the carbon dioxide gives the percentage of oxygen in the flue gases. Tubes *l* connect the pipettes.

30. The next operation is to drive the gas into the pipette *j*, Fig. 2, and then pass it into the pipette *i* to absorb any hydrochloric-acid fumes that may have been given off by the cuprous-chloride solution, if old; such fumes would increase the volume of the gases and make the reading on the burette less than the true amount. The sum of the percentages by volume of carbon dioxide (CO_2), oxygen (*O*), and carbon monoxide (*CO*) is subtracted from 100, and for practical purposes this difference is taken as the percentage by volume of nitrogen (*N*).

The gas must be passed through the burettes in the order named as the pyrogallol solution will absorb carbon monoxide and the cuprous-chloride solution will absorb oxygen. As the gases in the flue are under less than atmospheric pressure they will not of themselves flow through the pipe connecting the flue to the apparatus. The gas may be drawn into the pipe in the way already described, but this is tedious. A rubber bulb aspirator *g* connected to the air outlet of the cock *e* by tube *f*, will quickly draw a new supply of gas into the pipe. Another form of aspirator draws the gas from the flue in a constant stream, thus insuring a fresh supply for each sample.

31. The analysis made by the Orsat apparatus is volumetric; if the analysis by weight is required, it can be found from the volumetric analysis as follows: Multiply the percentages by volume by the molecular weight of each gas, and divide the products by the sum of all the products; the quotients will be the percentages by weight. For most work the use of the even values of the molecular weights insures sufficient accuracy.

The even values of the molecular weights of the gases that appear in an analysis by an Orsat are:

Carbon dioxide (CO_2).....	44
Carbon monoxide (CO).....	28
Oxygen (O_2).....	32
Nitrogen (N_2).....	28

Table V indicates the method of converting a volumetric flue-gas analysis into an analysis by weight.

TABLE V
CONVERSION OF A FLUE-GAS ANALYSIS BY VOLUME
TO ONE BY WEIGHT

Gas	Analysis by Volume Per Cent.	Molecular Weight	Volume Times Molecular Weight	Analysis by Weight Per Cent.
Carbon dioxide (CO_2).....	12.2	$12+(2\times 16)$	536.8	$\frac{536.8}{3022.8} = 17.7$
Carbon monoxide (CO).....	.4	$12+16$	11.2	$\frac{11.2}{3022.8} = .4$
Oxygen (O_2).....	6.9	2×16	220.8	$\frac{220.8}{3022.8} = 7.3$
Nitrogen (N_2).....	80.5	2×14	2254.0	$\frac{2254.0}{3022.8} = 74.6$
Total.....	100.0	3022.8	100.0

From the flue-gas analysis, the weight of air actually used for combustion can be computed from the following formula provided the percentage by weight of C in the fuel is known:

$$\text{Weight of air} = 3.036 \left(\frac{N}{CO_2 + CO} \right) \times C$$

In which N , CO_2 and CO are percentages by volume in the flue gas and C is the percentage by weight of carbon in the fuel.

The quantity of heat (B. t. u.) lost in the flue gases per pound of fuel is $L = .24 W (T - t)$.

In which W = weight in pounds of flue gases per pound of dry fuel.

T = temperature of flue gases, °F.

t = temperature of atmosphere, °F.

.24 = specific heat of flue gases.

The weight W of the flue gases per pound of dry fuel is computed from the analysis by the formula

$$W = C \left(\frac{11CO_2 + 8O + 7(CO + N)}{3(CO_2 + CO)} \right)$$

in which CO_2 , O , CO , and N are the percentages by volume, by analyses of the flue gas, and C is the percentage by weight of the carbon in the dry fuel.

The quantity of heat (B. t. u.) lost per pound of fuel burned through incomplete combustion of carbon and the presence of CO in the flue gas is in B. t. u. obtained by using the formula

$$L = 10,150 \times \left(\frac{CO}{CO + CO_2} \right) C$$

in which CO and CO_2 are the percentages of the gases by volume in the flue gases and C is the percentage of C by weight in the fuel.

ANALYSIS OF LIQUORS

32. Analysis of Sulphite Liquor, or Cooking Acid.—The liquor used in the paper industry for cooking wood chips in the sulphite process is variously termed sulphite liquor, sulphite acid, bisulphite liquor, cooking acid, or simply acid. Chemically, it is a solution of calcium and magnesium bisulphites in water with a considerable amount of free sulphur dioxide or sulphurous acid always present, and also small percentages of calcium and magnesium sulphates.

There are two general classes of tests for this important liquor: (1) rough mill tests, and (2) laboratory analytical

tests. Each has its proper function in the scientific control of the paper industry.

Rough Mill Tests.—The smell of the liquor is very unreliable in the case of an inexperienced operator, and quite reliable in the case of an experienced man. Consequently, no confidence can be placed in such a method of testing, and when it is found in use, it should be replaced with a simple mill test such as the following:

Take 1 cubic centimeter of the liquor and dilute with water to 250 cubic centimeters in a casserole or large evaporating dish. Using starch as an indicator, titrate with $n/10$ iodine solution. This gives the total sulphur dioxide SO_2 . Titrate a 1-cubic-centimeter sample with $n/10$ sodium hydroxide, using phenolphthalein as indicator; this gives the free sulphur dioxide, SO_2 . The difference between these two determinations gives the combined sulphur dioxide.

33. When this test is to be used frequently, and by workmen as well as chemists, it is well to prepare a chart from which the percentage of sulphurous acid corresponding to any given number of cubic centimeters of caustic soda or iodine can be read: Or, in some mills, the normality of the iodine and caustic-soda solutions is regulated so that a direct reading can be had. For instance, if the standard solution is made exactly $n/16$ instead of $n/10$, and a 2-cubic-centimeter sample of liquor is taken, the burette reading will give the percentage of sulphur dioxide SO_2 directly by moving the decimal point one place to the left. These methods all assume the specific gravity of the acid to be 1, which is naturally incorrect, but the error introduced in this way is not sufficient to vitiate the results for practical purposes.

Laboratory Method for Analysis of Sulphite Liquor.

1. The specific gravity may be determined with a pycnometer or with a Westphal balance.

2. Total sulphurous acid (SO_2): Take a 10-cubic-centimeter sample of the acid liquor, and dilute to 100 cubic centimeters exactly in a volumetric flask at room temperature. Take a 10-cubic-centimeter fraction of the diluted acid and titrate

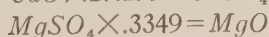
with $n/10$ iodine solution, using starch as an indicator. Multiply the number of cubic centimeters of iodine solution by .0032 and this will be the number of grams of sulphurous acid. Convert this into percentage.

3. Free Sulphurous Acid (SO_2): Take 25 cubic centimeters of the diluted solution prepared for the total sulphurous acid and titrate with $n/5$ sodium-hydroxide solution, using phenolphthalein as an indicator. Multiply the number of cubic centimeters of the sodium-hydroxide solution used by .016 to give grams of sulphurous acid. Convert into percentage.

4. Sulphates: Measure out 25 cubic centimeters of the original undiluted solution and add concentrated hydrochloric acid and boil until all the sulphurous acid is expelled, finally adding a slight excess of hydrochloric acid. To the solution, while still hot, gradually add barium-chloride solution (10 per cent.) Boil for about an hour; allow to settle, filter, wash, ignite, and weigh as barium sulphate. Calculate to sulphuric acid (SO_3) by multiplying the weight in grams by .343. The weight of calcium sulphate can be obtained by multiplying the weight of SO_3 in grams by 1.7.

5. Lime and Magnesia: Pipette out 25 cubic centimeters of the original solution; add about 1 cubic centimeter of concentrated sulphuric acid, evaporate to dryness in a platinum dish, igniting carefully, and cool and weigh the mixed sulphates as calcium sulphate and magnesium sulphate.

34. Dissolve the residue in 25 cubic centimeters of dilute hydrochloric acid, washing the solution into a beaker, and making it alkaline with ammonia. Heat to boiling and add enough ammonium-oxalate solution to completely precipitate the lime. Continue the boiling for two minutes, and let the precipitated calcium oxalate settle for one half hour. Filter, wash with hot water, ignite in a platinum crucible over a blast lamp to constant weight and weigh as lime. Calculate the lime to calcium sulphate, deduct from the mixed sulphates and calculate the magnesium sulphate to magnesium oxide.



6. Very divergent views are held as to the proper strength of the acid and the proper proportions of free and combined SO_2 . It used to be thought that acid should have a high combined SO_2 content. A usual analysis in the old days was 4 per cent. total SO_2 , 2.75 per cent. free and 1.25 combined. In such an acid only 70 per cent. of the total is free. Modern acid makers like to have anywhere from 78 per cent. of the total acid in free form.

35. It is now generally considered good practice to use an acid, with wet wood and a cook of 10- to 10½-hours duration, analyzing 4.65 per cent. total, 3.50 free and 1.15 combined; for dry wood, total 4.39 per cent., free 3.25 and combined 1.10 to 1.15. By *wet wood* is meant wood containing approximately from 40 per cent. to 50 per cent. moisture, and by *dry wood* is meant wood containing 27 per cent. to 35 per cent. moisture. The above strengths are the strengths of the cooking acid. The strength of the raw acid is about 3.60 per cent. total, 2.18 per cent. combined and 1.42 per cent. free in the case of the wet wood; and 3.86 per cent. total, 2.37 per cent. total free and 1.49 per cent. combined in the case of the dry. This acid is made at about 4.5° Bé., the strength being brought up to the total mentioned with reclaimed acid. For cooking bleached stock, stronger acid with a total of about 6 per cent. is preferable. The foregoing figures are for unbleached stock, and are all based on the use of the 1-inch chips, which run from 55 to 60 per cent. within this specified length; and it is also assumed that dry steam is supplied, generated at 150 pounds boiler pressure and with a pressure of from 60 to 68 pounds of dry steam on the digesters.

36. Strong acid means a higher sulphur consumption. Modern reclaiming systems make possible the maintenance of acid strengths that would have been out of the question before such systems were used, but with the best reclaiming systems, there is a direct relation between the sulphur consumption and the strength of the acid. For one thing it is not always possible to relieve all of the gas to the reclaiming system before blowing. In one mill, the conditions are such

that the digesters have to be blown very light to keep the temperature down. This involves blowing at a time when the acid tests 1.6 per cent. free, which means blowing sulphur into the air at the rate of about 150 pounds per ton of pulp. Systems for reclaiming the fumes from the blow pits have been devised to take care of just such cases as this.

Theoretically, 190 pounds of sulphur should be required to make 1 ton of spruce sulphite pulp. Most American mills use much more, some as much as 300 pounds. Certain Scandinavian mills make good pulp, using less than 190 pounds.

37. Analysis of Caustic Liquor.—Caustic liquor is used for digesting the wood in the soda process. The strength of this liquor varies in different mills from 11° to 12° Baumé at 60° F. In order to get good results, the liquor should be from 93.5 to 94 per cent. caustic; that is, this percentage of the soda present should be caustic. This test is practically the only one made.

Determination of Causticity.—Make up a weak solution of sulphuric acid (about 6 cubic centimeters of strong acid to 1 liter of water is a convenient strength). Take about 5 cubic centimeters of the caustic liquor (the amount should be varied according to the strength, but should never be so great as to require more than 50 cubic centimeters of the acid), dilute in a small beaker to about 50 cubic centimeters, and add a few drops of phenolphthalein. Titrate with the dilute sulphuric acid until the pink color just leaves, and take a reading. Now add a drop of methyl-orange solution, titrate cautiously until a slight pink tint is reached, and take a second reading. Subtract the first reading from the second, multiply the difference by 2, and subtract from the second reading. This remainder divided by the second reading and the result multiplied by 100 will give the percentage of causticity.

ILLUSTRATION.—The first reading when the pink color disappeared was 42.6 cubic centimeters. After adding methyl orange and titrating again until pink, the reading was 43.9 cubic centimeters. Following out the calculations as explained, $43.9 - 42.6 = 1.3$; $1.3 \times 2 = 2.6$; $43.9 - 2.6 = 41.3$; $41.3 \div 43.9 = .9407$; $.9407 \times 100 = 94.07$, which is the percentage of causticity.

38. Another method that gives very accurate results is as follows: Measure out 25 cubic centimeters of the caustic liquor and titrate with a normal acid, using methyl orange as an indicator. This will give the total alkali. Take 100 cubic centimeters of the same liquor and precipitate the carbonate and sulphate with neutral barium chloride in slight excess. Dilute to the mark in a 200-cubic-centimeter flask, shake well, and allow to settle. Take 50 cubic centimeters of this clear solution and titrate with the normal acid, using phenolphthalein as an indicator. This will give the amount of $NaOH$ in 25 cubic centimeters of the caustic liquor. The number of cubic centimeters of acid used in the second titration divided by the number of cubic centimeters used in the first titration and multiplied by 100 will give the percentage of causticity.

The liquor should also be tested for salt, especially where caustic liquor from an electric bleach plant is used in the liquor. This is done by titrating 5 cubic centimeters of the liquor with silver nitrate, using potassium chromate as an indicator.

39. Analysis of Black Liquor.—In the case of black liquor, it is at times desirable to know the strength both in total alkali and in free caustic acid. For total alkali, evaporate a measured volume to dryness in a platinum dish and ignite over a flame till the organic matter is completely carbonized. Cool, extract several times with hot water, and pour the extracts through a small platinum cone. Put the cone in the dish together with the residue of wet carbon, cover with a filter paper, which just fits inside the dish and quickly ignite over a gas flame. The filter paper prevents loss by spattering and enables the carbon to be burned off without waiting for it to be dried first. Again cool and extract with hot water, add the extract to the first and titrate with acid in the presence of methyl orange. Calculate the results to grams of sodium carbonate per liter, or pounds per gallon.

40. For the determination of free caustic soda in black liquor, add 25 cubic centimeters of the latter to 400 cubic centimeters of water and 15 cubic centimeters of barium-

chloride solution (400 grams per liter) in a beaker. Titrate directly with standard acid, using a dilute solution of phenolphthalein on a spot plate as an indicator. The acid should be added slowly and the end point may be considered as that point at which no pink color develops within 2 minutes after mixing 1 or 2 drops of the liquid with the indicator. Owing to the presence of soluble coloring matter and to the precipitate thrown down by the barium chloride, the end point is not very sharp; but with a little practice it is fairly easy to get concordant results. A sharper end point is obtained by filtering off, or settling out, the barium precipitate and using only the clear liquor, but this introduces a distinct error on account of the loss of the caustic soda occluded by the precipitate. In the method outlined above, this is kept within the sphere of action of the acid, and more accurate results are obtained.

41. Analysis of Sulphate, or Kraft-Process, Liquor.

The analysis of liquors for the sulphate process is much more complicated than for the soda or sulphite processes. The principal constituents of the liquor, however, are sodium hydroxide ($NaOH$), sodium carbonate (Na_2CO_3), and sodium sulphide (Na_2S). For the usual purposes of sulphate-mill control, it is sufficient to express the soda and sulphur contents of the liquor and ash in terms of the above.

1. *Determination of Sodium Sulphide.*—Take a cubic-centimeter sample of the liquor and dilute to 100 cubic centimeters. Take 10 cubic centimeters of the diluted liquor and add a known excess of $n/10$ iodine solution; titrate this with $n/10$ sodium-thiosulphate solution, using starch as an indicator. Calculate to Na_2S .

2. *Determination of Na_2S , $NaOH$, and Na_2CO_3 Together.*—Take 10 cubic centimeters of the diluted liquor prepared for 1, and add $n/10$ sulphuric acid until all the hydrogen sulphide is discharged. Test this by odor, and by a strip of filter paper moistened in lead-acetate solution. If the hydrogen sulphide is not entirely discharged, the lead acetate paper will turn black. Titrate the excess of sulphuric acid with standard

alkali, using phenolphthalein as an indicator. The number of cubic centimeters of standard alkali used will permit the calculation of the original alkali present determined as Na_2S , $NaOH$, Na_2CO_3 .

3. *Determination of $NaOH$ and Na_2S .*—Take 10 cubic centimeters of the diluted liquor as prepared for 1, and treat with 10 per cent. barium-chloride solution. Titrate with $n/10$ sulphuric-acid solution.

The content of the liquor can be calculated from the foregoing determinations very readily. 3-1 gives $NaOH$; 2-3 gives Na_2CO_3 ; and 1 itself gives Na_2S .

TESTING OF WOOD PULP

COLOR VALUE OF PIGMENTS

42. The price of a pigment does not always indicate its value. This is determined by the coloring power of the pigment. A chemical analysis will determine the value of a pigment, but there are simple methods of comparing colors and dyes that can be applied by persons not having a knowledge of chemistry.

Comparison of Pigments.—To compare two pigments, weigh out 1 gram of the higher-priced one and mix with 10 grams of a good, dry, china clay. Weigh out as many grams of the lower-priced pigment as can be bought for the price of 1 gram of the higher-priced one, and mix with 10 grams of the same clay. Mix separately in a mortar with water to a thin, pasty mass. Spread each upon a strip of glass side by side and dry in a steam bath. The colors can now be compared, and the pigment having the higher color value selected. This test may be carried still further by weighing out a larger amount of clay and adding to the one giving the strongest tint in the preceding test until it matches the other. By noting the difference in the amount of clay used, the difference in value of the two pigments can be determined.

Comparison of Soluble Colors.—All soluble colors, such as aniline colors, are dissolved in the proportion of 1 gram to the liter. Some colors have greater affinity for paper fibers than have others; also, some colors have greater affinity for one kind of fiber than they have for other kinds of fibers. In order to compare such colors in regard to their relative coloring power, the comparison must be made on the same paper stock, or combination of fibers, on which the colors are intended to be used.

43. If, for instance, it is desired to compare the relative strength of two aniline blues for use in coloring or whitening newspaper made up of 75 per cent. of ground spruce wood and 25 per cent. of spruce sulphite fiber, then some newspaper stock made up of those two pulps in the proportions given should be used in making the comparison. This is done as follows:

A quantity of the pulp is taken, thinned with water, and thoroughly mixed to a uniform mixture. The dilution should be such that 200 cubic centimeters will contain about 1 gram of dry fiber. Of this thinned pulp, exactly the same volume (200 cubic centimeters) is measured out for each color to be compared. These portions are poured into white-china bowls. With a pipette, the same measured amount of each color solution is run into each of the bowls. In the case of acid colors, alum is added, the same quantity of alum solution being added to each vessel, so that everything connected with each test is the same. In case of basic colors, no alum is added, as none is required; in fact, alum may prove injurious to colors of this kind. The mixtures of pulp and color are stirred from time to time and allowed to stand for about $\frac{1}{2}$ hour. By this time the colors have gone on to the fibers to their maximum extent. The contents of each bowl is now poured on a wire cloth of fine mesh, and the water draining off, leaves a sheet of colored paper formed on the wire. A sheet of muslin is spread over this sheet of wet paper, and a sheet of blotting paper is then placed on this muslin and pressed so as to absorb some of the water from the wet paper. When

the muslin is stripped off, it carries the sheet of colored paper with it. This is next pressed between two fresh pieces of muslin and blotting paper, and the sheet of colored paper is then stripped off from the pieces of muslin and hung up to dry. When dry, the sheets are compared as to the strength or depth of color. All conditions being identical, the color that produces the strongest coloring effect is, of course, the strongest color.

44. In order to tell just how much stronger one color is than another, the same measured quantity of the same thinned pulp used in the first comparative test is taken, and 5 per cent. less of the stronger color than was used in the first test is added. To another measured portion 10 per cent. less color is added; to another 15 per cent. less; and so on, until a sheet is obtained that is colored to exactly the same extent as with the weaker color. This, of course, shows how much stronger the one color is than the other.

In order to determine whether a dry, powdered, soluble color is a straight color or a mixture of two or more colors, a very simple test is to wet a piece of filter paper and then blow some of the powdered color on it. Where each speck of color strikes the wet filter paper, it colors the paper with its own individual color. For example, suppose it is desired to learn whether a red-shade blue color, such as is ordinarily used for newspaper, is a straight blue of uniform red shade or a mixture of a blue-shade blue with some red color. By blowing some of the powder on a wet sheet of filter paper it will be found that if the color is straight, each particle will produce the same kind of blue spot on the wet paper, but if the color is mixed, there will appear both blue and red spots. It is sometimes preferable to wet the filter paper with alcohol instead of water, as the former dissolves some colors better than the latter and consequently the colored spots show up better.

45. Determination of Cellulose in Wood.—In attempting to determine the percentage of cellulose in any kind of wood, it is especially important that the samples should

be selected in such a manner as to be truly representative. They should be taken from sound, clean wood, free from knots, blemishes, or bark. Samples of wood should be clamped in a vice and sufficient powder rasped off with a coarse file from which to make an analysis. Naturally, if the wood in question is being used for making ground wood, the analysis can be made satisfactorily with a sample of the ground-wood pulp. The method to be subsequently given is that in use in the laboratory of Arthur D. Little, Inc., and was invented by Arthur D. Dean, and has been found to give satisfactory results on a great variety of woods.

46. Add 200 cubic centimeters of 1-per-cent. sodium-hydroxide solution to the fiber, cover with a watch glass and boil gently for $\frac{1}{2}$ hour, washing the fiber down from the sides of the vessel several times. Filter with suction on a 1-inch perforated plate placed in a 5-inch funnel. A little of the fiber will run through the plate at first and must be poured back after a good mat, like an asbestos filter, is formed. In order to hold the plate firmly in the funnel during filtration and the subsequent manipulations, pass a piece of fairly stiff silver wire up through one of the holes in the center of the plate and securely fasten it by bending the end down. Let the silver wire extend down through the stem of the funnel, projecting $\frac{1}{8}$ inch beyond the end. By putting one or two slight bends in this wire, it can be made to bear against the inside walls of the stem holding the filter plate firmly in place.

47. Wash the boiled wood with a good volume of hot water, suck dry, loosen up the fiber with a sharp-pointed glass rod and attach the stem of the funnel to the rubber tube leading from a chlorine generator (under the hood). Cover the funnel with a watch glass and pass a stream (about 1 or 2 bubbles a second) of washed chlorine gas up through the fiber. Continue the chlorination for 1 hour. Every 15 minutes, the fiber should be loosened and the lumps broken up with the pointed rod.

48. After chlorination, return the funnel to the suction flask and wash with hot water to remove *HCl*. Place 150

cubic centimeters of 2 per cent. sodium carbonate in a wash bottle. Invert the funnel over a 500-cubic-centimeter casserole, push on the silver wire until the main mass of the fiber with the filter plate drops into the casserole, turn the funnel right side up and wash all the fiber with a stream of sodium-sulphite solution from the wash bottle. Wash the wire and plate and add the remaining sodium carbonate. Bring the mixture to a boil and boil for 5 minutes. Again collect the fiber on the filter plate, wash with hot water until the washings are colorless, loosen up, and expose to chlorine as before.

49. With the wood of some broad-leaved species, all of the lignin is removed by the first chlorination and, instead of coloring yellow the second time in chlorine gas, the fiber bleaches to a pure white. With most coniferous woods, the fiber turns yellow when exposed to chlorine the second time. In that event, chlorination is continued for $\frac{1}{2}$ hour, the fiber washed and boiled in alkaline, sodium-sulphite solution as before, and again exposed to chlorine. Except in very unusual cases, the fiber will bleach when exposed to chlorine the third time. At whatever stage the fiber bleaches white, remove it immediately from the generator, wash on the filter plate with a large amount of water, transfer with the aid of distilled water to a casserole, let stand under water for a short time, collect in a large-sized, tared, Gooch crucible, and wash well with alcohol and finally with ether. Dry the product at first at a gentle heat and then weigh it.

It is almost impossible to wash out the last traces of acid, and unless the fiber is washed with alcohol and ether, the slow drying with the concentration of acid at the drying surfaces will cause a browning of the edge of the cellulose. If, for any reason, there should be any mineral matter present which would not be removed by the treatment, ignite the dried product and subtract the weight of the ash, to obtain pure cellulose.

ANALYSIS OF PULP

50. Determination of Moisture in Wood Pulp.—Wood pulp is offered for test under such a variety of conditions and in so many different forms that no one method of sampling and testing can be made applicable to all forms; for example, the strip method of sampling, while well adapted to mill sampling, is not suitable for the sampling of frozen lap pulp at receiving point; the auger method, while well adapted to the sampling of baled pulp, is not suited to the sampling of wet lap pulp. Neither is the wedge method, nor any other method which involves the breaking open of the bales, suitable for referee sampling of pulp at dock, since transportation companies decline to handle broken bales, or bales which have not been rebaled as perfectly as they were at point of shipment, which is impossible with ordinary hand presses.

At the outset, it may be stated that accuracy in sampling depends more upon taking sufficient samples from individual units, whether bales or laps, to insure a fair average of the lot, rather than upon any particular method of cutting the samples.

Forms in Which Wood Pulp is Presented for Test.—The forms in which wood pulp is available for test include the following: Baled pulp—dried sheets, hydraulic pressed laps; roll pulp; loose lap pulp—wet laps, hydraulic pressed laps; wet machine pulp in sheets. The official testing of pulp is also governed to some extent by the place, time, and quantity of pulp to be tested.

Determination of Wet Shipping Weight of Pulp.—As all pulp is invoiced on the basis of air-dry tons, rules for the determination of the wet weight of the pulp are just as important as rules for sampling and testing, and the wet weight should be determined by one of the following methods:

1. Railroad weight of entire car lot where tare of empty car is actually determined by weighing. This does not mean the routine bill-of-lading, railroad weight, but an actual weighing upon railroad scales supervised by party or parties at interest.

2. Weight of entire car lot as certified by official recognized weighing bureau issuing weighmaster's certificate of weight.

3. Wet weight of lot by sum of weight of truck loads passing over accurately tested scales during loading or unloading.

4. Wet weight of lot may also be determined by multiplying the actual number of bales or rolls, as determined by accurate count, by the average weight of at least 5 per cent., preferably 10 per cent. or more of the normal bales, or 20 per cent. of the rolls, selected for weighing. Where the bales are numbered, the numbers upon the bales selected should be indicated in the report.

Definition of Normal Bale.—A normal bale should be intact and unbroken. Reject as abnormal those bales, the weight of which exceeds by 50 pounds, or falls below 50 pounds of mill standard weight. For example, if the standard bale weight is 450 pounds, reject as abnormal those bales exceeding 500 pounds and those falling below 400 pounds in weight. With half bales, those weighing approximately 250 pounds, reject as abnormal those bales exceeding 275 pounds, and those falling below 225 pounds.

Recognized Methods for Sampling Pulp.—The first recognized method for sampling pulp is known as *strip method* .1. In this case, the pulp is tested, or sampled, at the pulp mill, and the varieties of pulp suitable for sampling by this method are: (a) Pulp coming from wet machines; and (b) pulp coming from driers in continuous web.

Cut a 2- or 3-inch strip across the entire width of the web coming from the machine. The rate of sampling wet machine pulp is one sample as just described for every 2,000 pounds of production. The rate of sampling the web-dried pulp is one sample from at least every fifth bale, or roll, of production.

It is to be noted that where this method is applied to dried pulp, the net weight of shipping units should be taken at substantially the same time as sampling for the moisture test.

In case the pulp is stored at the mill before shipment, the shipping units, lap, rolls, or bales, shall be subject to methods of sampling covering pulp in these various forms. The wet weight of the lot is to be determined at the time of sampling.

The second recognized method for sampling pulp is known as *strip method B*. In this case, the pulp is sampled in the store room of the mill, and the varieties suitable for sampling by this method are: (a) Pulp coming from the wet machines; (b) pulp suitable for unpressed lap pulp; and (c) roll pulp.

Take a 2- or 3-inch strip of wet machine pulp through the center of the sheet alternating with and across machine run. The sampling is to be continuous through the lot and to give at least 2 kilograms of wet sample per standard 30-ton car.

For lap pulp unpressed, take a 2-inch strip through the center of the lap, alternating the long way and the short way of lap. The depth is half way through. The sampling is to be continuous through the car lot and to give at least 2 kilograms of wet sample per standard 30-ton car.

For roll pulp, take a test strip 3 inches in width across the face of the roll from the second layer and four similar strips from layers located at least $1\frac{1}{2}$ inches from the outside layer.

For the third, or *auger method*, the varieties used in testing, or sampling, are: (a) Baled pulp in sheets; (b) baled hydraulic-pressed laps; and (c) rolled pulp.

The sample should be taken by boring into a bale to a depth of 3 inches (7.62 centimeters), with a special auger which cuts a disk about 4 inches (10.16 centimeters) in diameter. The disks shall be removed and ten of them taken as a sample, these to be selected as follows: One disk second sheet from the wrapper. Two disks 1 inch (2.54 centimeters) deep. Three disks 2 inches (5.08 centimeters) deep. Four disks 3 inches (7.62 centimeters) deep.

The holes to be bored shall be so located that in 5 successive bales they will represent a portion extending diagonally across the bale. Each bale is to be bored but once. The first bale is to be bored at the corner, the edge of the cut being at a distance of 1 inch from the edge of the bale. The second cut shall then be made half way between the location of the first cut and the center of the bale; the third bale shall be cut at the center; the fourth bale half way between the center and the corner, and the fifth bale in the opposite corner in a position corresponding to the first.

Not less than 5 or 10 per cent. of the entire shipment, and not less than 10 bales, shall be sampled. The samples shall be drawn from only sound and intact bales selected from different sections of the entire shipment. The analyst shall be careful to observe that no unusual conditions prevail in the selection of bales.

In the fourth, or *wedge method*, only two varieties of pulp are suitable for sampling, (a) lap pulp, and (b) hydraulic-pressed laps.

The angle of the wedge is exactly 9 degrees. A wedge sample is to be cut from every one hundredth lap when piled loose in the car. The wedge is to be approximately $2\frac{1}{2}$ inches at its widest point and is to extend to center of lap. The depth of the cut is to half through the lap. The cuts start

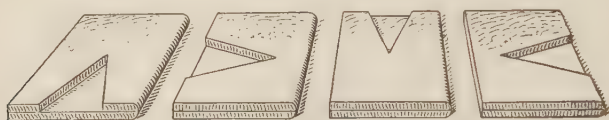


FIG. 3

at the middle of the closed edge of the lap as first position and then successive laps will be cut going around clock-wise, as shown in Fig. 3.

Care of Wet Samples.—The samples, as taken, are to be deposited immediately in a metal can with a tight-fitting cover, and the net wet weight obtained as soon as practicable after taking the samples. In the case of unavoidable delay between the time of taking the samples and in weighing, a special precaution should be taken to seal edges of cover air-tight with friction tape, or otherwise.

Weighing of Samples.—Accurate scales and weights to be used for weighing both wet and dry samples. Both wet weight and dry weight should be obtained upon same scales with same weights. Hot, dry samples should not be exposed to air during weighing.

Sensibility of Scales.—Scales used for weighing pulp samples must show a sensibility of .1 per cent. of maximum load. For example, if 1 kilo of pulp samples is taken, the scales

must show a decided deflection by the addition of 1 gram to load.

Drying of Samples.—After weighing wet samples, these should be placed in a suitable oven and dried to constant weight at 100° C., or 212° F. Maximum allowed variation from this temperature at time of final weight, plus or minus, should not exceed 5° C. For determination of final dryness, two successive weighings should not show a variation greater than .1 of 1 per cent. of wet weight of samples, and the minimum of these weights is to be taken as final bone dry. To calculate bone dry to air-dry basis, divide percentage of bone dry by .9 to give the percentage of air dry.

PAPER TESTING

TESTING FOR QUALITY

51. Samples of paper are tested in the same way as pulp for moisture and ash, taking about 50 grams for the determination of moisture and 3 or 4 grams for the ash.

Microscopic Fiber Test.—The various fibers in paper are detected by means of a microscopic examination, while at the same time there are several chemical tests that are useful in identification.

Prepare the sample to be examined under the microscope as follows: Cut small pieces of paper from different parts of the sample and boil in a 1-per-cent. solution of caustic soda. The fibers may now be separated by shaking in a bottle containing a few pieces of broken glass. The fibers are placed on a glass and covered with a drop of glycerine and a cover glass.

Mechanical Wood Fiber.—Under the microscope, *mechanical wood fiber* is distinguished from *chemical wood fiber* by the fact that the fibers are rarely separated, they being generally bound together in bundles. The ends also are torn and jagged.

Linen Fiber.—In Fig. 4 is shown an illustration of *linen fiber*. The ends of this fiber are usually drawn out into

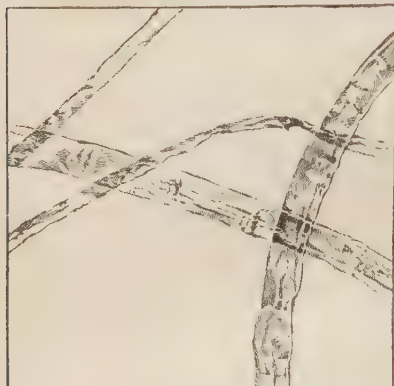


FIG. 4

numerous fibrils and fibers of a cylindrical form.

Esparto and Straw Fibers.—The esparto and straw fibers are very much alike, and consist of serrated cells and fibro-vascular bundles. Straw may be distinguished from esparto by the presence of small, oval-shaped cells and the absence of the fine hairs that always line the inner surface of the leaf of the esparto plant.

Chemical Wood Fiber.—The fibers of chemical wood are flat and ribbon-like and have unbroken ends, very much like cotton, but are distinguished from it in that they are not twisted. Pine-wood fiber is detected by the presence of small pitted vessels.

Cotton fiber is shown in Fig. 5, poplar fiber in Fig. 6, and spruce fiber in Fig. 7.



FIG. 5

52. To assist in the determination of different fibers under the microscope, a reagent made up as follows will be found of great help: Dissolve 2.1 grams of potassium iodide and .1 gram of iodine in 5 cubic centimeters of water and mix with 20 grams of zinc chloride dissolved in 10 cubic centimeters of water. The mixture should be

allowed to stand and the clear liquid decanted. This reagent will color cotton, linen, and hemp, wine red; esparto, straw, and wood cellulose, bluish violet; mechanical wood pulp and unbleached jute, yellow; and manila hemp, blue and bluish gray to yellow.

Chemical Tests for Constituents of Paper.—When a small quantity of paper that has been beaten up, separated, and moistened with iodine is examined under the glass, the fibers can be separated, owing to the fact that the ground wood and jute will be colored yellow; cotton, hemp, and linen, brown; while bleached straw, esparto, and chemical wood fiber will remain uncolored.



FIG. 6

Detection of Mechanical Wood Pulp.—Nitric acid gives a brown stain on paper containing mechanical wood pulp.



FIG. 7

Phloroglucinol gives a deep magenta in the presence of mechanical wood pulp. The latter test is the one mostly used for this purpose. Phloroglucinol is prepared by dissolving 2 grams of the reagent in 25 centimeters of 95-per-cent. alcohol and adding 5 cubic centimeters of concentrated hydrochloric acid. Apply a drop to the paper to be tested and allow to evaporate.

In the presence of ground-wood pulp, the deep magenta coloration will be developed.

53. In some papers, the coloring matter used acts as an indicator for acids and will turn red when treated with acid. In papers where these colors are present, it is difficult to test for ground wood with phloroglucinol, as the acid in the mix develops the red color. In papers where yellow mix is used, this color is developed by the acid in the phloroglucinol. Aniline sulphate develops a deep yellow in presence of ground wood, and is prepared by dissolving in water. This reagent should be substituted for phloroglucinol in tests where colors such as those just mentioned have been used in the paper.

The percentage of ground wood may be roughly estimated by comparing the depth of color produced by the phloroglucinol on the sample with the depth of color produced on several standard samples in which the percentage of ground wood is known. This test can be applied only when the chemical fiber present has been perfectly reduced.

Detection of Sulphite Pulp.—Sulphite pulp can be detected by means of a dilute solution of sodium-auric chloride, which imparts to the paper moistened with it a reddish-brown color when unbleached sulphite pulp is present, and a bluish color when bleached sulphite pulp is present.

Detection of Straw and Esparto.—Straw and esparto can be detected by boiling the paper for some time in a 1-per-cent. solution of aniline sulphate, which produces a red color in the presence of these substances.

Detection of Animal Size.—Heat a small fragment of paper in a test tube with distilled water, transfer the clear solution to another tube, cool, and add a solution of tannic acid. If any animal size is present, a milky, flocculent precipitate will be formed, the consistency of which will depend on the amount of animal size present.

A very delicate test for animal size is to soak a small strip of paper in a reagent prepared by dissolving a small amount of mercury in an equal weight of fuming nitric acid, cooling, and adding an equal volume of water. In the presence of animal size, the paper will develop a red color, the depth of which will depend on the amount of size present. This reagent will keep only about a month.

Detection of Starch.—Starch may be detected in paper by adding a drop of very dilute solution of iodine, which, if starch is present, will develop a deep-blue color. This test is better carried out by boiling some of the paper in water for about 20 minutes, pouring off the water into another vessel, and adding the dilute iodine solution.

Detection of Rosin Size.—The presence of rosin size may be detected by heating some of the paper in absolute alcohol and pouring the alcoholic solution, after cooling, into five times its volume of distilled water. If a precipitate is formed or there is a cloudiness, the presence of rosin size is indicated.

By wetting the sheet with the tongue and holding it to the light, a well-sized paper will not be transparent, while if poorly sized, it will be transparent, the degree of transparency varying inversely with the amount of size used.

Detection of Chlorides.—Boil the paper in distilled water, filter, add a few drops of nitric acid, and then a few drops of silver-nitrate solution. A cloudiness or a precipitate indicates the presence of chlorides.

Detection of Alum.—An excess of alum can be detected by boiling a quantity of the paper in a small amount of water, filtering, and testing the filtrate for aluminum by precipitating with ammonium hydroxide and ammonium chloride.

The nature of the filler can be determined by an analysis of the ash.

Determination of Coloring Matters.—Sometimes, coloring matters can be determined by an examination of the ash. When ultramarine has been used, the ash is blue.

MECHANICAL OR PHYSICAL TESTING OF PAPER

54. Paper may be examined from three different points of view: *physical quality, mechanical quality, and composition.*

The various tests classified under physical testing are: weight, thickness, bulk, sizing, etc. The chief mechanical test is for strength, and this is subdivided into tests for tear, punch, bursting, etc. Under composition falls the determination of the materials used in making the paper, and their proportions.

In addition to the preceding tests, special tests may be used on special grades of paper, namely, absorbency and permeability for blotting papers, resistance to blood in butchers' papers, etc.

55. In most cases, not one of the foregoing tests will give an accurate working knowledge of the paper. Moreover, some one test may frequently show the paper to be of excellent quality, but when this paper is put to use it is found to be worthless; consequently the nature of the test used on the paper must be in accordance with the treatment the paper will encounter in actual use. For example, for testing strength there are several machines in general use which give the punch test, bursting test, tear test, etc. It is often quite desirable to have a sheet of paper comparatively low in punch test but high in tear, as, for example, a bag paper. A punch test applied to such a paper, or to any paper for that matter, shows the hardness and rigidity of the sheet.

However, the punch test is not what should determine the value of the bag sheet. A thin sheet of celluloid would test very high on such a machine and would have no value at all for bag manufacture, and this is only an extreme case of the conditions prevailing with some paper. Consequently, the sheet to be tested must be dealt with in such a manner that its tearing resistance will be shown and the extent to which the fibers will peel when they are torn apart. A wrapping, or bag paper, that peels when torn will be much better than one that does not. A punch test will not show this peeling quality at all. In fact, if two sheets of bag, or wrapping, paper were made, one stiff and hard with little crossing of the fibers, the fibers beaten short and the sheet well-colored and sized, and the other sheet made soft and flexible, with the fibers brushed out long and interwoven on the paper machine wire, the punch test would show the first sheet to be the better of the two, as it would give a higher test. In reality, however, the second, from the point of view of service, would be by far the better sheet, and a tear test would indicate this, as would also a peeling of the fibers.

56. The foregoing remarks will illustrate the necessity not only of making tests, but also of making the right kinds of tests.

Before making any physical test on a sample of paper, it is always advisable to determine what is known as the *machine direction* of the sample of paper. By this is meant the finding out which side of the sample, as cut, was running with and which was running across the web of paper as it came from the machine. This can frequently be ascertained by mere inspection of the sheet. The machine wire frequently imparts a wire mark to the paper.

If a piece of the paper is allowed to float on the surface of a vessel of water, it will usually roll up and the axis of the roll will indicate the direction in which the paper passed through the machine. If a Mullen tester is available, the machine direction can easily be found, as the chief line of rupture is always across the sheet.

The following are descriptions of some of the commoner paper-testing machines with some remarks as to the purposes for which they are useful.

Weighing Devices.—Paper scales of the beam type with a wire hook for holding the sheet of paper, are usually used for weighing full-sized sheets of paper. This type of scale is extensively used in paper mills and is usually graduated so as to give the weight of reams of 480 and 500 sheets. The weight of paper is usually expressed in pounds per ream of a given size.

The *Perkins paper scale* is of the quadrant type and is similar in principle to the ordinary letter scale used for weighing mail in business offices, only it is more delicately adjusted. This scale permits of the direct reading in pounds of the weight per ream of 500 sheets.

Thickness Gauges.—Thickness gauges are micrometers which by means of a system of springs and levers register on a dial graduated in thousandths of an inch. Such instruments usually get out of order and should be tested from time to time with a set of standard sheets of material ranging from .001 to .015 inch.

Schopper Tester.—The Schopper testing machine measures the tensile strength of paper and other materials. In this device a strip of paper 15 mm. (approximately $\frac{1}{2}$ inch wide) by 180 mm. long (approximately $7\frac{3}{8}$ inches) is clamped at each end and the clamps are moved apart until the strip is broken. A suitable device indicates the pull in kilograms (approximately 2.2 lb.) required to break the strip. It is recommended that the load in kilograms per 15 mm. width strip, be converted into pounds per inch of width by the following formula:

$$(3.73) \times \text{tensile strength in kg. per 15 mm. width} = \text{tensile strength in pounds per 1 inch width}$$

The *Mullen tester* measures the bursting strength of paper. By means of a hand wheel the hydraulic pressure is increased on a column of glycerine which drives a small rubber ball through the sheet of paper, which is tightly held between two flat surfaces. Pressure is read on a dial graduated so as to give the bursting strength in pounds per square inch. The operation of the Mullen machine is very simple and tests can be made very quickly. Consequently, it has been very widely adopted throughout the paper industry and, for some grades, the Mullen test is really a very fair criterion of the strength or the particular variety of strength required. However, as previously explained, for many kinds of paper, the Mullen test is of very little value and is frequently positively misleading.

The *District of Columbia paper tester* is very similar in construction, and operates on the same principle as the Mullen tester.

Folding Machines.—For many kinds of paper it is especially important to determine the fold endurance. This is usually measured by machines of the *Schopper folding-machine* type, which folds the paper backwards and forwards until it is worn through.

57. The folding strength of paper is dependent not only upon the strength and durability of the paper, but also is very largely influenced by the stretch of the paper and by relative

humidity. To perform this test in the most accurate manner, it is necessary to keep the relative humidity constant for all tests. This can be done only by the use of a room where the humidity is under control. Where such a room is not available, attention should be paid to the per cent. of relative humidity of the air at the time of the test. No tests should be made when humidity is either very high or very low. A relative humidity between 65 and 70 per cent. is most easily attained through the year and is the standard humidity recommended.

The calibration and standardization of the folding tester require much attention, since there are several moving parts that become worn. To check the results obtained on different machines, it is necessary to check the bearings, rollers, and the tension of the springs carefully, and have their dimensions all accurately the same.

Tear-Testing Machines.—For many kinds of paper as, for instance, in bag and wrapping papers, the tear test is of the utmost importance.

In the *Thwing machine*, a small sample of paper is cut and punched by a special die and attached to two pins, one of which is attached to a movable weight on an arm carrying a recording pen, while the other is attached to a motor-driven, sliding, record-card holder. This holder is caused to move away from the pin on the weight arm, thus tearing the paper along the line of perforations. The record shows graphically the force in grams required to tear the paper between each two perforations, thus giving five peaks, the average of which is taken as the tearing strength.

The *Witham tear-testing machine* imitates the tear given to a sheet of paper by the human fingers, but in such a manner that the weight necessary to effect this tearing is automatically registered accurately down to small fractions of a gram. The paper is torn to a certain distance whereupon a round hole is punched in the sheet, insuring an absolutely fair start or coefficient for the tear. This is important because a sheet of paper partially torn will allow the tear to continue with much less stress than would be required to start the tear in the first place.

The *Widney testing machine* is a very useful machine for determining the stiffness, resilience, and point of yield in any pliable material. The tearing resistance of paper can be definitely determined in ordinary units. This machine is especially applicable to heavy papers, boards, etc., where stiffness is one of the most important properties.

The *Webb tester* is a testing machine of the puncture type, which gives more satisfactory results on many materials than the Mullen tester and which can be used for certain products such as fiber board, for which it is not possible to adapt the Mullen machine. In addition to the puncture test, Webb machines may be used for tensile tests, elongation tests, and compression tests. While its use was developed in connection with the testing of fiber boards and corrugated fiber containers, it can also be used for testing any kind of paper, cardboard, building boards, etc.

The *Schopper tensile-strength machine* has also been used for this purpose and has been found to give fairly concordant results, provided that the speed of tearing is kept constant.

Absorption and Sizing Tests.—The absorption and sizing tests have not been thoroughly standardized, but the following are those in use in one of the largest and most successful paper-testing laboratories in America.

The test for *writing papers* is to float a piece of paper 1 or 2 inches square on the surface of ink, and note the time in minutes until the ink begins to be visible on the upper surface. The time in minutes is taken as a basis for comparing the relative sizing of different papers.

To avoid discrepancies due to the variations in the ink, it should be made according to the following formula:

Gallotannic acid.....	23.4 grams
Gallic acid.....	7.7 grams
Gum arabic.....	10.0 grams
Hydrochloric acid.....	2.5 grams
Ferrous sulphate crystals ($FeSO_4 \cdot 7H_2O$)	
.....	30.0 grams
Methylene blue...	2.0 grams
Water to make up 1 liter	

Allow to settle for several days and then decant for any sediment.

For all papers other than writing papers use the so-called *ferrocyanide test* as follows:

58. Float a piece of paper about 2 inches square on the surface of a 5 per cent. solution of potassium ferrocyanide, and note the time. Then test the upper surface of the piece of paper from time to time by stroking (across the machine direction) with a small camel's-hair brush moistened with a solution of ferric chloride (5 to 10 per cent.). When the ferrocyanide has soaked through the paper sufficiently to come in contact with the ferric chloride, it will react the moment the latter is applied and it will give a blue color. The penetration is then considered complete, the time is again noted, and the length of time since the paper was laid upon the surface of the solution is taken as the measure of its resistance to penetration. In stroking the paper with a camel's-hair brush, take care to select a place on the paper which has not previously been wet with the ferric-chloride solution. Report the results to the nearest minute, or, if the time is very short, in seconds.

The absorption test on *blotting paper* is carried out as follows: Cut strips of paper, in both directions, about .5 inch wide and at least 3.5 inches long, and make a pencil mark about $\frac{1}{4}$ inch from one end. Then, starting from this mark, make a series of pencil marks at intervals of $\frac{1}{8}$ inch on the paper for a distance of about 2.5 inches. Suspend the strips in water in such a manner that the surface of the water coincides with the first mark. When the strip is first placed in the water, note the time and at the end of 3, 5, and 10 minutes, respectively, ascertain the rise of the water in the paper by means of the marks upon it. Report the results in sixteenths of an inch; that is, a paper which shows an absorption of 1 inch will be reported as Absorption, 16. If necessary a small quantity of ink may be added to the water to aid in determining the exact rise of the liquid. Test several strips of each sample cut from each direction and report the average absorption.

CONSIDERATION OF WATER USED

59. Water Supply.—In consequence of the very large volumes of water required in pulp and paper manufacture, an adequate supply of water of a suitable degree of purity is a matter of prime importance. The quantity of water used will vary greatly with the kind of paper made. In a mill making fine paper from rags, where the stock is washed for a long time with a great deal of water, volumes of water will be required that are enormous in comparison with the output of the mill. In mills making newsprint, wrappings, etc., the washing is not so elaborate, but the greater volume of material handled makes the volume of water very large. The volume of water used in making a ton of paper is probably never less than 50,000 gallons and is sometimes as much as 200,000 gallons.

A mill making 80 tons of bag and wrapping paper each day, uses approximately 15,000,000 gallons of water per day for all purposes. In another mill making no pulp, but only paper from rags, purchased pulp, and other materials, the water consumption is approximately 4,500,000 gallons per day. As the mill turns out only about 25 tons of finished product per day, this is a large consumption of water, and is accounted for by much washing of rags, bleach stock, etc. If there is any impurity in the water, it will get into the paper, since the fibers in the forming web of paper on the wire form a regular filter that will catch and retain any sediment or coloring matter.

Two kinds of coloration are in water. Coloration due to sediment, which can usually be removed by settling, or by coagulation with alum or sulphate of iron in settling tanks, or ponds; and dissolved color, usually resulting from decaying vegetable matter, which is very hard to remove.

Hard water is water containing mineral salts in solution which are chiefly lime salts, the carbonate being the most common of these. Rain water is perfectly soft, but as it falls through the atmosphere, it dissolves carbon dioxide and this enables it to dissolve lime and other minerals out of the soil and rocks over which, or through which, it runs. Thus, almost all water taken

from streams, lakes, wells, etc., needs to be softened before using.

In pulp and paper mills, objection is made to hard water on two general grounds: First, it should not be used in the boilers; and, second, it is unsuitable for use in the actual manufacture of pulp and paper.

Considering the second point first, if hard water is used in the beaters, an excessive amount of alum will have to be added, in order to get rid of the hardness, because no size will be precipitated by the alum until all hardness is removed from the water. Not only does this use up alum, but it also imparts an element of uncertainty to the whole sizing operation, since the hardness of the natural water will vary from day to day and the beaterman will never know how much alum should be added. Frequently, beatermen will attribute this trouble to variations in the chemical strength of the alum, but usually the water is at fault.

Hard water is undesirable for washing any kind of pulp, either after manufacture, or after bleaching. In fact, it is much better if the mill has a uniform supply of soft water so that no hard water need be used at any stage of the process. Since a water-softening system ought to be installed for the use of the boiler house, it is a comparatively easy matter to make it at the same time of sufficient capacity to serve the whole mill.

60. Treatment of Boiler Feedwater.—It is not possible within the scope of this work to enter into detail on the treatment of boiler feedwater, a subject which would require a complete treatise in itself. It is necessary, however, to touch on the great importance of this matter as a problem in practical pulp and paper operation. Preparation of water for boiler feed purposes falls naturally into two main classifications: One is the removal of sediment and mechanically suspended material of all kinds; the other is the softening of the water, or the removal of dissolved salts of lime, magnesium, etc., which tend to incrust or corrode the boilers and, in general, to lower their efficiency.

Hardness in boiler feedwater is the direct cause of scale, leaky flues, muddy burning, and many of the other troubles incident to large steam production. It shortens the life and reduces the efficiency of the boiler, increases the repair expense, and frequently causes inopportune and costly delays and shutdowns. Most harmful of all, the scale, acting as a thick and effective insulation of the flues, causes an enormous increase in the consumption of fuel. The fuel wastage due to the use of hard water may be anywhere from 10 to 30 per cent.

61. The cost of boiler cleaning, boiler repairs, and boiler compounds, etc., all caused by the use of hard water, is also a big item in the operation of a large plant. Boiler compounds should be viewed with suspicion. They are only temporary makeshifts at best. A steam boiler is not designed as a vessel in which to carry out chemical reactions, and the constant adding of materials to the water in the boiler cannot help but corrode and weaken the boiler itself. The aim should be to supply as nearly as possible pure water to the boiler at all times. The purification of the water should be effected in a purification and softening plant of sufficient capacity, outside the boiler system.

PURIFICATION SYSTEM

62. Water purification systems mostly fall into three classes. These are (1) lime process; (2) soda process; and (3) lime and soda process.

Lime Process.—The carbonates of lime and magnesia, to which the hardness of the water is due, are precipitated with slaked lime in solution (lime water) in some suitable form of apparatus, either with or without heat.

Soda Process.—Soda ash (sodium carbonate) and caustic soda (sodium hydroxide) are added either alone or together to the water to be softened. Caustic soda is more efficient in this process than soda ash. This treatment is used with water containing sulphates of lime and magnesia. Barium

hydrate is sometimes used instead of caustic soda, or soda ash, or in certain proportions with them.

Lime and Soda Process.—When both sulphates and carbonates of lime and magnesia are contained in the water to be softened, this process is used. The soda is added first, enough being used to consume all the sulphates present, and then lime is added.

Simple boiling will soften water if it is merely afflicted with *temporary hardness*, that is, contains carbonates of lime and magnesia. If the water possesses *permanent hardness*, that is, contains sulphates of lime and magnesia, mere boiling is of no avail.

Zeolite Process.—There is one other excellent method of softening water, the zeolite system. This system avails itself of the properties of an interesting material known as zeolite, which automatically removes calcium salts from the water, yielding perfectly soft water from any ordinary source. In time, the zeolite becomes exhausted, but can easily be renewed by a simple treatment. This system is in successful use in many industrial plants. It is a rather expensive system for use simply on boiler feedwater, but is ideal where perfectly soft water is needed, as in textile manufacturing and certain branches of paper manufacture.

WATER TROUBLES

63. Besides hardness there are some other troubles with water, which are important from the power-plant point of view. The first one of these is *acidity*. This may be due to industrial waste dumped into streams by chemical plants, tanneries, textile plants, smelters, other pulp mills, etc. In sulphite mills, there is always a possibility, if check-valves do not work properly, or in the event that indirect heating equipment for digesters becomes leaky, of acid getting into the boiler feedwater. This causes acid corrosion, which creates more havoc even than scale. If there is any possibility of acid getting into the boiler feedwater, very frequent tests should be made with litmus, or an electric device may be arranged which

will ring a gong as soon as there is enough acid content in the boiler feedwater for a current of fixed intensity to pass through the water, the conductivity of which is increased by the acid. Such devices are much used in Sweden and Finland where indirect cook is much used in the digester house.

Foaming.—Microscopic organisms, sewage, industrial wastes, etc., are the chief substances present in water which cause this trouble. Filtration, sometimes preceded by the addition of a coagulating agent such as alum or sulphate of iron, is the usual cure for this evil.

Priming.—Priming is the giving off of steam in spurts and belches, caused by excessive amounts of salts in solution in the boiler feedwater. Too much soda, added to get rid of scale-forming impurities, is a frequent cause of this condition. Frequent blowing down of the boiler is the usual remedy. If this has to be resorted to at too frequent intervals, it is advisable to look about for some new source of water supply.

64. Any and all of the systems of water softening may be preceded by a system for clarifying or removing suspended matter and sediment. This usually consists of filter beds, or tanks, the use of a coagulating substance, such as alum or ferrous sulphate (sulphate of iron), being now quite general. Alum, in addition to removing sediment, will frequently remove dissolved colorations.

None of the preceding systems should be confused with water treatment for sanitary purposes. Water may be frequently crystal clear and also perfectly soft by chemical test, and, therefore, entirely suitable for boiler or for making paper and yet contain large numbers of bacteria of the most virulent nature. To purify water in the sanitary sense, as is done by towns and cities, extensive filtration, accompanied by treatment with chlorine, or bleaching powder, is needful.

WATER FOR FIRE PROTECTION

65. So far much has been said about the quality of water and nothing about the quantity. The water supply of a pulp and paper mill should be adequate to take care, of not only

the normal needs, but also of the requirements, in case of fire. There should be a storage tank of adequate capacity mounted on a tower sufficiently high to give a satisfactory nozzle pressure at the top of the highest building or wood pile in the plant. The water supply should be connected with the supply of the town, city, or village (assuming that the plant is situated near one) or of some other nearby, large, manufacturing plant, so that assistance can be rendered in case fire or explosion puts the power plant out of commission. Conversely, this enables the pulp and paper mill to be of mutual service in case of emergencies.

66. Assuming that a pulp-and-paper industry consists of a ground-wood mill, a sulphite mill, and a paper mill, should each of these have its own water supply, or should there be a single pumping installation? This will depend entirely on local conditions, the distance separating the various parts of the plant, etc. This is typical of the engineering questions concerning paper mills about which no general directions can be given. Neither can anything definite be stated with regard to the kind of pump to be used for general water supply. Both centrifugal and plunger pumps are used, multistage turbine pumps being very generally satisfactory.

MANUFACTURE OF SUGAR

(PART 1)

INTRODUCTION

1. Definitions.—The term **sugar** was originally employed and intended to classify all substances having a sweet flavor, and thus came to be used almost indiscriminately for cane sugar, fruit sugar, lead acetate (so-called sugar of lead), and other compounds possessing this property. At present, in a general sense, it is reserved almost exclusively to denote crystallized sugar from the tropical cane and the sugar beet, which is *sucrose*, or *cane sugar*. The commercial sugar, whether derived from the tropical cane, the sugar beet, the maple tree, the palm, or some other source, is almost pure sucrose.

2. In chemistry, the word sugar is applied to a large group of compounds of carbon termed *carbohydrates*. These bodies are subdivided into four groups, as follows: (1) **Monosaccharides**, which among other sugars include the glucoses, $C_6H_{12}O_6$, dextrose (also called grape sugar and starch sugar), and levulose (fructose, fruit sugar); (2) **disaccharides**, or sugars, the most important of which is sucrose, or cane sugar, $C_{12}H_{22}O_{11}$; (3) **trisaccharides**, of which raffinose, $C_{18}H_{32}O_{16}$, is of analytical importance, as it occurs in small quantities in the sugar beet; and (4) **polysaccharides**, or starches, $(C_6H_{10}O_5)_x$.

3. The glucoses, dextrose and levulose, are monosaccharides of the hexoses. They occur naturally in plants and are also formed by the hydrolysis of cane sugar. The hydroly-

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ysis is usually accomplished by the action of dilute acids or by ferments on the sugar in water solution. The process is often termed *inversion*. The products of the inversion are equal quantities of dextrose and levulose, and the mixture is called *invert sugar*.

As the word indicates, *hydrolysis* means an absorption of water. Thus, when sucrose is subjected to hydrolysis, the reaction indicated by the following formula occurs:



Chemists have not succeeded in accomplishing the reverse of this reaction; that is, the production of cane sugar from the glucoses.

It will be noted in the study of the processes of manufacture that precautions are always observed to avoid, so far as possible, the hydrolysis, or inversion, of the sucrose by the action of acids or otherwise.

4. History of Sugar.—Etymologically, sugar would seem to be of Indian origin, the earliest forms of the word being *çarkarā* in Sanskrit and *sakkara* in Prakrit. Thence it may be traced through all the Aryan languages, as *shakar* in Persian, *sakkar* in Arabic, *suicar* in Assyrian and Phenician, *saccharum* in Latin, *azúcar* in Spanish and Portuguese, *zuchero* in Italian, *sucre* in French, *zucker* in German, etc.

The precise product indicated by these various names is not always clear, and probably is not identical in all cases. The cultivation of the genuine sugar cane (*Saccharum spp.*) appears to have been common in India and China in very remote times, but there is no documentary evidence on this point earlier than Herodotus. Frequent mention of the "sweet cane" occurs in the Scriptures, but the plant referred to is doubtful. An Indian reed yielding honey is alluded to by Strabo, and a similar statement concerning an Egyptian reed is made by Theophrastus; while Dioscorides actually gives the name *saccharum* to a kind of honey obtained from reeds in Arabia Felix and India; both he and Pliny accurately describe the product as being white and brittle and of a salt-like consistency. Later it seems to have been generally

termed *Indian salt* among the Greeks and Romans, by whom it was obtained in small quantities at great expense from India, and used medicinally.

5. The introduction of cane sugar in the Mediterranean basin must have occurred at an early date; for it was found growing at Assouan, on the Nile, in 714, and was carried into Spain by the Moors in 766, while Sicily engaged in the culture about 1060 to 1090. During the religious wars of the middle ages, the "sweet honeyed reeds," called *zucra*, which abounded in the meadows about Tripoli, were consumed by the Crusaders; and it is evident that sugar making in that neighborhood was conducted in a wholesale and systematic manner. From Cyprus and Madeira, the industry extended, in 1500 to 1600, to most of the West Indies, where it was carried on by Spanish and British colonists; but there is strong evidence in favor of the supposition that several kinds of sugar cane are indigenous both to the West Indies and to almost the whole continent of South America.

6. **Establishment of Refineries.**—From the extensive growth of sugar in the western tropics there ensued large importations of the raw article into Europe. The introduction of tea and coffee about the same time created a general and wide demand for what had hitherto been regarded as a medicine rather than a nutritive article of diet. Sugar refining appears to have been copied from the Arabs by the Venetians. Refineries were established in England and Germany in the 16th century, and in Holland soon after, and utilized the imported raw sugar.

7. **Sugar From Beets.**—Up to this time sugar derived from cane was the only kind known in commerce. But, in 1747, Margraf, a German chemist, demonstrated the existence of about 6 per cent. of sugar in beet root; and in 1795, Achard manufactured beet sugar on his farm in Silesia, Germany, and presented loaves of refined sugar to Frederick William III, of Prussia, in 1799. About ten years later, Napoleon used extraordinary efforts to foster the production of native-grown sugar, and grapes, plums, maize, sorghum,

carrots, etc. were also experimented on. The results obtained did not excel those from beets, and the first French factory for making beet sugar was established at Lille, in 1810, by Crespel-Delisse. The sudden and great fall in the price of sugar, caused by the declaration of peace between France and Germany, in 1815, crippled the native industry in Germany as well as in France, but a few of the manufacturers held on tenaciously, and the production of beet sugar, in France alone, rose through many vicissitudes from 1,000 tons in 1827 to 1,075,000 tons in 1906. In Germany, the production in 1906 amounted to 2,400,000 tons.

8. Sugar Production in the United States.—Prior to 1889, the sugar produced in the United States was almost exclusively derived from the tropical cane that grew in Louisiana. The refining branch of the industry depended on foreign sources for nearly its entire supply of raw sugars, which were derived from both beet- and cane-sugar producing countries. Since 1889, and especially since 1899, there has been a large production of beet sugar in America. This sugar is usually ready for consumption when it leaves the factory. The Louisiana factories formerly produced sugars ready for immediate consumption, but at present raw sugar for refining is manufactured almost exclusively. Other American sources of sugar are the maple tree and sorghum. The production from these two sources, however, especially the latter, is of little commercial importance.

9. Chief Sources of Sugar.—From the preceding statements, it may be seen that the world's sugar supply is obtained almost exclusively from two plants—the sugar cane and the sugar beet. Only a comparatively small quantity of palm sugar is produced, and the small quantity of maple sugar made in America is consumed in the raw state.

CANE SUGAR

SUGAR CANE AND ITS COMPOSITION

10. The Plant.—Sugar cane, *Saccharum officinarum*, is a very large grass; its stalk is round and jointed. Normal canes are never hollow. The exterior color varies with the variety. The stalks of the varieties usually grown for sugar production are of a greenish yellow, a purple or purplish-red color, or have stripes of red or purple and green. In certain varieties the green is so light that the canes are often spoken of as being white. The height to which the cane grows varies greatly with the soil and climatic conditions, and ranges from 2 to 6.5 meters (about 7 to 20 feet), seldom attaining the latter height. The cane grown in Louisiana and Texas never reaches full maturity; it attains a height of from 2 to 3 meters (about 7 to 10 feet). The Cuban cane, under favorable conditions reaches a height of from 3.5 to 4 meters (about 11 to 13 feet). The leaves of the sugar cane are ribbed, and, in different parts of the plant, their length ranges from a few centimeters to 2 meters (from about 1 inch to about 7 feet). Sugar cane is a tropical plant, though it is grown successfully in subtropic regions, notably in Louisiana and Texas. Outside of the tropics, it rarely reaches full maturity and produces seed.

11. For commercial purposes, cane is always grown from cuttings, the plantlets sprouting from the eyes at the joints and never from the seed. The stalks reserved for planting are popularly termed "seed." Until within recent years, the true seed of sugar cane was supposed to be infertile. The discovery of canes that were apparently seedlings led to investigations that proved that a part of the seed is fertile. Large experimental plantings of seedlings have been made

with a view to improving the sugar-producing qualities of the cane, and as a result of many comparative trials a few new varieties have been selected and introduced that are large sugar producers. Canes grown from cuttings have all the characteristics of the original plant, while those from the seed usually differ greatly from the parent. So far as is now known, cuttings from all parts of the stalk, when planted, produce canes of equal sugar-making qualities.

12. Planting of Sugar Cane.—The seed bed is usually prepared with great care in Louisiana and in other localities where the conditions are favorable to thorough plowing. This is not the case in many parts of the tropics, and especially in recently cleared lands that cannot be prepared for plowing except at an enormous expense. Where the conditions favor it, the cane is planted in shallow furrows, entire stalks or cuttings being used as seed. In order to secure a good stand of cane, two parallel lines of stalks, laid breaking joints, are often planted. This method of planting in furrows is usually adopted where cane is cut only twice from one planting or where the lands are old. In Spanish-America, especially in recent clearings or in comparatively new lands, the usual method is to plant in small trenches, each about a yard long and a yard apart in the row, a single short cutting of cane being planted in each trench. The cane rows are usually about 6 feet apart.

Cane may be planted at two seasons of the year, namely, spring and fall; or, if in the tropics, at the beginning of the rainy season and near its end. The cane is called *spring* or *fall plant*, according to the time of planting. Whether planted in clumps or in a furrow, the cane soon forms a compact row if the soil is good. A new crop of cane springs up from the stubble left after harvesting, and in the tropics, many crops may be harvested from a single planting. Usually, only two crops are harvested in Louisiana from one planting.

13. Harvesting of Sugar Cane.—Full maturity of the cane is never awaited before beginning the harvesting. It is

essential that the factories have a long working season; hence, just as soon as the stalks contain sufficient sucrose to justify sugar manufacturing, the cutting of the cane begins. The cane reaches sufficient maturity in Louisiana from about October 20 to November 1, and in the tropics from about 6 to 8 weeks after the close of the rainy season. The cane is at its best in Cuba after March 1, and at this time, in favorable localities, the juice contains about 18 per cent. of sucrose and has a coefficient of purity of about 88°, although these figures are often exceeded. The juice from Louisiana cane, even when at its best, does not often contain so much as 15 per cent. of sucrose, and the average analysis for an entire crop seldom shows more than 13.5 per cent. of sucrose.

14. In harvesting the cane, the stalk is first stripped of its leaves by two or three deft strokes with a cane knife having a broad blade; it is then cut down close to the ground, and finally the top is removed at the highest joint that is sufficiently matured. The joint at which to top the stalk is determined by a change to a lighter color. This joint is easily selected in red or stripped canes, but not readily with the extremely light-colored varieties. In Cuban practice, the canes are subdivided into lengths of about 1 yard so as to facilitate loading. After cutting, the canes are thrown into rows or piles, and later they are loaded in carts or cars and hauled to the factory. The loading is usually accomplished by hand, but during recent years mechanical loaders have come into use.

The cane should be ground as soon as possible after it is cut down. This is especially the case in the tropics, where there is always a rapid fall in the coefficient of purity of the juice, with a loss of sucrose, when cut cane is exposed to the sun.

The canes produced from the seed, or cuttings, are termed *plant canes*, and those from the stubbles are called *ratoons*, or *stubble cane*. The ratoons are better sugar producers than plant canes.

The harvesting season lasts from 10 to 12 weeks in Louisiana, and usually about 5 months in the tropics.

15. Composition of Cane and Cane Juice.—The composition of the cane is extremely variable and is influenced largely by the variety, the soil, the climate, and the age of the plant. Stubble canes, or ratoons, usually contain more sugar and woody fiber than plant canes. According to Dr. C. H. Browne, Jr., of the Louisiana Sugar-Experiment Station, the composition of the purple variety of Louisiana cane, as indicated by the results of many analyses at the time of harvesting, in November–December, is as follows:

	PER CENT.
Water	74.5
Ash5
Fiber	10.0
Sugars { sucrose dextrose levulose }	14.0
Nitrogenous bodies4
Fat, wax, pectin (gums), free acids, combined acids6
Total	100.0

The reaction of the cane juice with litmus is always strongly acid.

The preceding analysis refers only to that part of the cane harvested for sugar making. The unripe top joints of the stalk are always left in the field.

16. The juice of the cane varies greatly in composition with the part of the stalk from which it is expressed and with the pressure exerted in extracting it. Compared with the upper part, the lower part of the stalk contains a relatively high percentage of sucrose and low percentages of dextrose and levulose. In addition to the sugars, the juice contains mineral (inorganic) salts, nitrogenous bodies (albuminoids, etc.), and non-nitrogenous bodies (wax, fat, etc.).

The sugar content of the juice varies considerably, even in canes grown in the same district. The age of the cane, the

cutting, that is, whether plant cane or ratoons, the soil, the elevation and drainage of the land, and the character of the fertilizers, if used, all affect the sugar content of the juice. Excessive use of a nitrogenous fertilizer produces a rank growth of cane, having a comparatively low sucrose content and an impure juice; whereas, a phosphatic fertilizer is favorable to the production of rich cane with juice of high purity. Cane grown on low land usually contains less sugar and juice of lower purity than that grown on higher lands in the same vicinity.

Mature cane of a good variety contains a high percentage of sucrose and a very low percentage of dextrose and levulose. In such canes, the levulose is often present as a faint trace, or is absent, and the dextrose amounts to only one-tenth or two-tenths of a per cent. In some rare cases, the only sugar present is sucrose.

The relation between the sucrose and the non-sucrose solids of the juice obtained by milling is modified greatly by the amount of pressure applied and the use or non-use of water to saturate the residue of the cane after the first grinding. The juice obtained by light pressure contains less non-sucrose in proportion to the sucrose than that resulting from heavy pressure or from heavy pressure combined with water saturation of the cane residue. Notwithstanding the lower purity of the juice, that is, regarding everything present except sucrose as an impurity, the modern conditions in sugar production demand the highest possible yield of juice in the milling process.

17. Sugars of the Cane.—Except under very favorable conditions of culture, and then only for a short period, the cane contains three sugars, namely, *sucrose*, or *cane sugar*, *dextrose*, and *levulose*. In the cane-sugar industry, the dextrose and levulose are usually grouped together and termed glucose, or invert sugar, the former term being generally employed. The presence of other sugars has been surmised by some investigators. From the manufacturing point of view, however, only the three sugars mentioned are of prime

importance, and a knowledge of their properties will therefore be of great practical value to the sugar maker and the chemist.

18. Sucrose, or Cane Sugar.—The sugar known as **sucrose**, or **cane sugar**, forms hemihedral crystals of the monoclinic system. These crystals are transparent and are readily soluble in water and in dilute alcohol. Absolute alcohol, anhydrous glycerine, ether, and chloroform have only a slight, if any, solvent effect on sucrose.

This sugar is rapidly acted on by mineral acids and more slowly by organic acids. This action, as previously mentioned, is termed inversion, or hydrolysis, and the product, consisting of a mixture of equal parts of dextrose and levulose, is called invert sugar. The invert sugar, as well as each of its constituent sugars, is directly fermentable, but sucrose is not. Sucrose must therefore be converted into invert sugar, either by the action of acids or by certain ferments, before it can be fermented.

Sucrose may be inverted by heat alone when in water solution and more rapidly in the presence of air. From this it may be seen that the loss of sugar was great in the old methods of manufacture, in which the juice and concentrated liquors were boiled in the open air. Since the boiling point of the liquid is raised as its concentration increases, the loss of sucrose by inversion is greatest in boiling heavy sirup in an open pan. In modern methods, the liquors are neutralized and concentrated in vacua, thus partly or entirely eliminating losses by inversion from heat and acids. Inversion may occur through the heating of sugar solutions in the presence of inorganic salts and the salts of organic acids, but in the manufacture it is probable that the losses of sucrose from these sources are small. Sucrose, when moist, decomposes at a temperature of 100° C. The heating surfaces of evaporating apparatus should therefore always be kept covered with the sugar solution.

19. Dextrose.—The sugar called **dextrose** forms rhombic crystals when anhydrous, and the hydrated sugar

forms transparent crystals, or crusts. Dextrose is soluble in water and in alcohol, its solubility in the latter depending on the concentration of the solvent.

20. Levulose.—The sugar called **levulose** forms colorless, needle-shaped, rhombic crystals. These crystals are very hygroscopic and are very soluble in water and in alcohol. It is extremely difficult to produce these levulose crystals, and for this reason and owing to the hygroscopic properties of this sugar, invert sugar is frequently termed *incrystallizable sugar*.

21. General Remarks on the Components of Sugar Cane.—A study of the properties of the sugars and other components of the cane is necessary to a clear understanding of reasons for the various steps in the manufacture of commercial sugar. It should be noted that sucrose in water solution is very readily inverted by heat and also by acids; that levulose is easily decomposed by heat and by alkalies; and that dextrose is also decomposed by heat in an alkaline solution, with the formation of highly colored bodies, but resists decomposition more than does levulose.

As will be shown later, both the application of heat and the use of an alkali are necessary in the purification of the juice. Knowing the properties of the sugars, it will be seen that in order to obtain a satisfactory quality and yield of sugar, all highly acid or alkaline solutions must be avoided in the processes of manufacture, as must also excessively high temperatures in treating or concentrating the saccharine solutions in the open air. Unfortunately, to produce the grades of sugar required by the markets, it is not always practicable to work under ideal conditions, with respect to the stability of the sugar. Owing to the tendency of the sugars to decompose and to ferment, cleanliness of tanks and floors is necessary and the work should be conducted with regularity.

The principal component of the juice that must be eliminated in the manufacture is the albuminoid matter. This matter is coagulated at a temperature of about 80° C., but

in the usual defecation process the temperature is raised above 90° C. The coagulated albuminoid matter is somewhat soluble in alkaline juice. As lime is used in the purification of the juice, it is obvious that the reagent must be used with caution.

The ash of the juice contains silica, potash, soda lime, magnesia, iron, phosphoric acid, sulphuric acid, and chlorine. Very little of these substances is removed in the processes of manufacture, and most of them pass into the final residue—the molasses. Part of the acids of the juice is precipitated by the lime in the defecation process, and part remains combined with the reagent in the form of lime salts. Part of the fat, wax, and gums is extracted with the juice in the milling process, and of these substances, the fat, the wax, and to some extent the gums are removed in the defecation process.

COEFFICIENTS USED IN SUGAR MANUFACTURE

22. For convenience and brevity, several expressions, or coefficients, are used by sugar makers and chemists in describing the condition of the juice and products at various stages of the manufacture. These coefficients may be defined as follows:

23. Coefficient, or Quotient, of Purity.—The coefficient, or quotient, of purity shows the relation between the sucrose and the total solids contained in the material; it is the percentage of sucrose in the total solid matter in the substance. In calculating the *apparent coefficient*, that is, the coefficient ordinarily employed, the solid matter in the juice or in a solution of the material, as indicated by a Brix hydrometer, and the sucrose, as estimated by a direct polarization, are used. The *true coefficient of purity* is the percentage of sucrose in the solid matter, the latter being determined by drying the material.

The true coefficient is used in comparative laboratory tests and in special researches but not usually in conducting the manufacture. The apparent coefficient is meant when the

word "true" is not prefixed, and usually only the word "purity" is used to designate this coefficient. Purities are commonly expressed with the degree mark instead of the percentage sign, or both signs are omitted.

24. The coefficient of purity of cane juice is considered very low when less than 80° , and very high when it reaches 90° . That of the final molasses product is very low in tropical factories at about 30° , and in Louisiana this number often falls below 25° . This difference between the tropical and Louisiana molasses purities is due to differences in the composition of the canes and possibly at times to a decomposition of sucrose.

From these remarks it may be seen that the coefficient of purity is indicative of the sugar-making qualities of the juice, since the higher the purity, the greater the proportion of sugar that must be removed to reduce the molasses to a given purity. In good work, with rich canes, 90 per cent. or more of the sucrose in the juice is recovered in the sugars. The final molasses usually varies but little in purity, whereas the coefficient of the juice often varies greatly. The significance of the coefficient of purity should be thoroughly mastered, since in modern factory methods all the work is conducted with regard to the purity of the materials in process.

25. Glucose Coefficient, or Glucose Ratio.—The **glucose coefficient** is the ratio of the glucose (invert sugar) to the sucrose, multiplied by 100. An increase in this coefficient from one stage of the manufacture to another is usually indicative of inversion of sucrose. This is not necessarily the case, however, since the sugars may not be removed in like proportions in the processes of manufacture, and the mechanical losses may not balance one another.

26. Saline Coefficient.—The **saline coefficient** is the ratio of the percentage of ash to that of the sucrose. It is little used in the cane-sugar industry.

27. Dilution.—By dilution is meant the quantity of water added to the normal juice in milling or in diffusion, to increase its weight to that of the diluted juice. The dilution is usually expressed in percentage terms of the weights of the cane and calculated water of dilution.

28. Saturation.—The quantity of water sprayed on the bagasse, in percentage terms of the weights of the cane and water, is the per cent. of **saturation**, or **maceration**.

MANUFACTURE OF CANE SUGAR

29. Synopsis of Manufacture.—The various stages of the manufacture of cane sugar may be briefly outlined as follows: (1) The extraction of the juice—(*a*) milling processes, and (*b*) diffusion processes; (2) the purification of the juice, that is, the defecation and clarification; (3) the concentration of the juice to a sirup; (4) the crystallization of the sugar—(*a*) crystallization in the vacuum pan, (*b*) crystallization at rest, and (*c*) supplemental crystallization with motion; and (5) curing the sugar—(*a*) purging in the centrifugal, (*b*) drying, or granulation, of the sugar, and (*c*) formation of cubes and loaves.

EXTRACTION OF JUICE

30. Processes.—Two processes are used in the extraction of juice from sugar cane, namely, *milling* and *diffusion*. A vast majority of the factories use mills, but very few employ diffusion batteries.

31. Milling Processes.—Mills are usually constructed of three heavy cast-iron rollers that are fitted with strong steel shafts and heavy gearing and housings. The arrangement of a mill with a cane shredder is shown in Fig. 1, and with a Krajewski crusher in Fig. 2. At the left of each of the figures is the mill and near the center and above it, in Fig. 1, is the shredder, and in Fig. 2, the crusher.

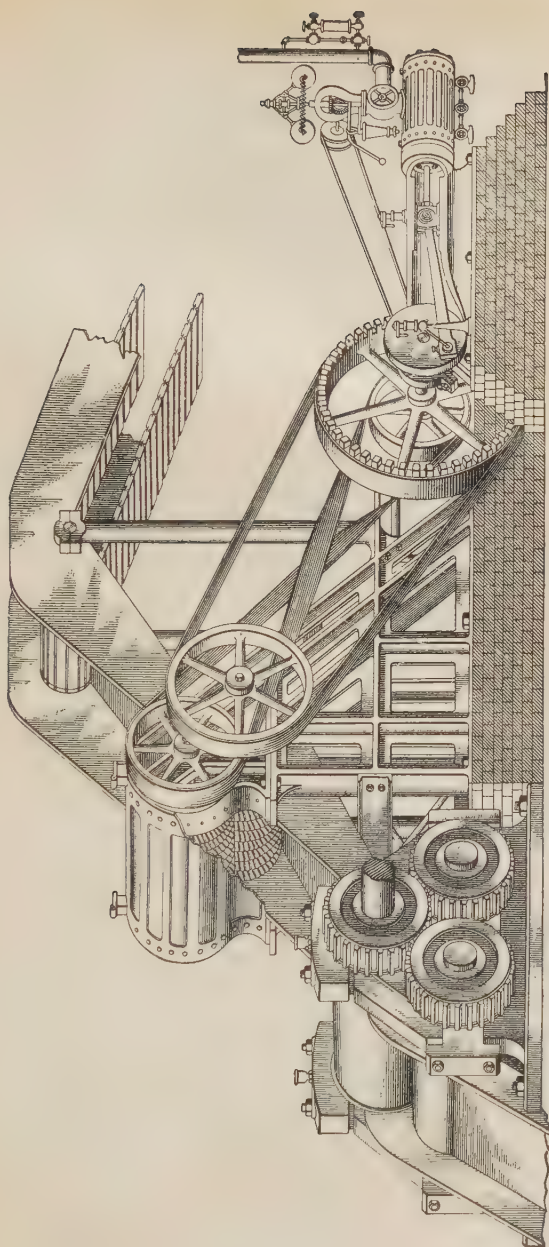


FIG. 1

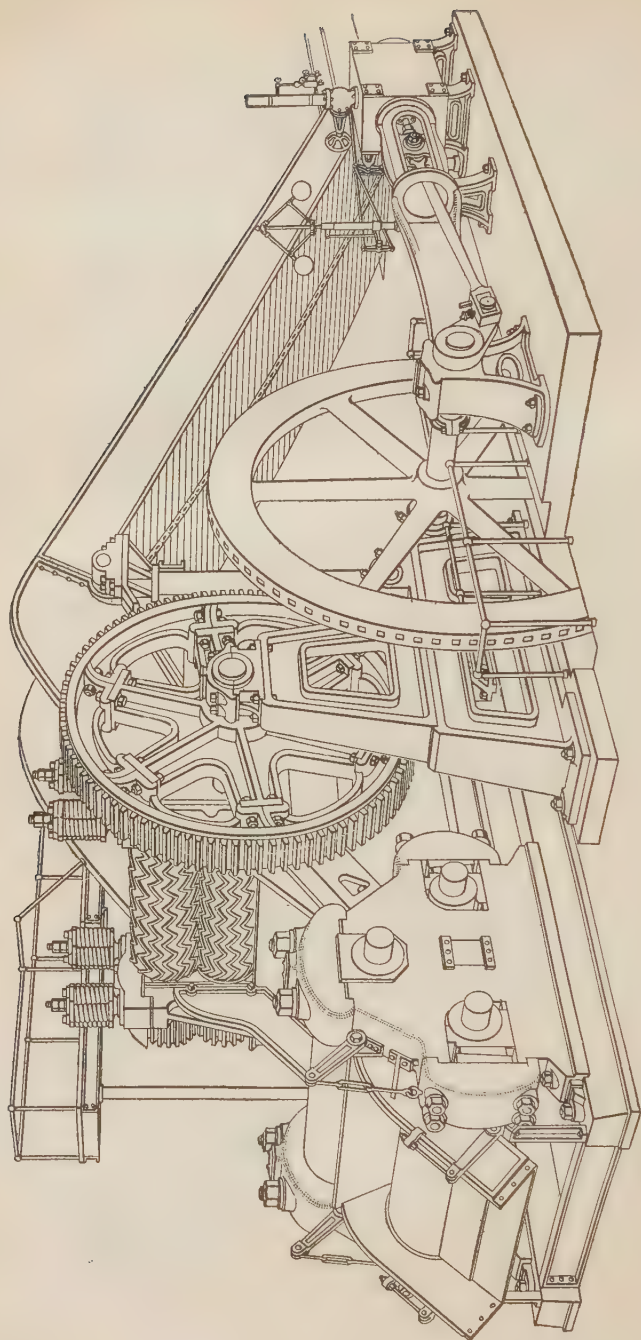


FIG. 2

A chute directs the cane between the top roller and the front, or cane, roller. The partly crushed cane is then forced over a heavy iron plate, called a *turn plate*, or *dumb turner*, and passes out of the mill between the top roll and the back, or bagasse, roll. By means of bolts, which show in the figures at the top and sides of the mill, the rollers are adjusted to give the desired pressure. The space left between the top roller and the cane roller is somewhat greater than that between the top and bagasse rollers. The setting of these rollers is determined by the strength of the mill and the number of mills worked in combination with one another. The pressure on the top roller is often regulated by a hydraulic device, which permits the roller to lift if the feed of cane becomes too thick, or if a piece of iron or other foreign body enters with the cane.

Other combinations of rollers than three, notably two rollers, are occasionally used, but modern plants almost invariably consist of one or more three-roller mills. Instead of using a single three-roller mill, the modern factories usually have two or three mills, and occasionally four mills, driven by one or two engines. The mills are set "tandem," that is, one after another, and the cane passes from mill to mill. The residue, or bagasse, from the last mill of the series is conveyed directly to the fires for the production of steam. This residue is termed *megass* by the English.

32. It is the usual practice to shred, or crush, the cane preliminary to grinding it. Two types of machines are very generally used for this purpose, namely, the *National cane shredder* (Fig. 1), invented by Samuel Fiske, of the Newell Universal Mill Company, the first successful preparatory machine; and the *Krajewski cane crusher* (Fig. 2), of the Krajewski-Pesant Company. The *Marshall crusher*, made by the Whitney Iron Works Company, which is similar to the Krajewski crusher, but differs in the shape of the teeth and certain other details, is also used to some extent.

33. The **National cane shredder**, as shown in Fig. 1, has two cutting, or tearing, rolls made up of toothed steel

disks. The disks of one roll enter the spaces between those of the other, as shown. The disks are driven in opposite directions and at different rates of speed, thus tearing the cane fed to them into shreds, and depositing the shreds in an even layer in front of the mill rolls. The juice that is separated is immediately reabsorbed by the shredded cane. This is not a crushing process.

The **Krajewski crusher**, as shown in Fig. 2, is designed to break the rind and partly extract the juice. The cutting, or crushing, rollers have sharp-edged, wavelike corrugations.

The object of the shredder or the crusher is to relieve the first mill of the strain of crushing the hard rind of the cane. This enables the mills to do more and better work. It also prepares the bagasse to receive water of saturation as will be described later. The crusher also extracts considerable juice.

34. The cane is usually conveyed to the mills on a moving platform called a *carrier*, or *conductor*. It is transferred from the carts or cars by hand or by mechanical devices. In several of the large plants of recent construction, the cane is hoisted from the cars in bundles of 4 or 5 tons and is dropped into a large hopper. Then it is conveyed to the mills by chains, equipped with strong fingers or arms.

The first mill is usually adjusted to extract juice to the extent of about 60 per cent. of the weight of the cane. The second and third mills are set to extract as much juice as their strength will permit. Under these conditions, the bagasse leaves the last mill with a water content of approximately 51 per cent. and containing from 8 to 10 per cent. or more of sucrose, these figures varying with the juiciness and richness of the cane. In order to reduce this great loss of sugar, the bagasse is usually saturated with water after it leaves the first or the second mill, thus diluting the juice it contains before regrinding. This process is termed *saturation*, *maceration*, or *imbibition*.

35. Two methods are commonly used in applying the saturation water, namely, *single* and *double saturation*.

In **single saturation**, the water is sprayed on the bagasse as the latter is expanding on leaving the mill. The water may be applied at the first or the second mill, or at both. The usual method is to apply the water to the bagasse leaving the second mill, as it is then in better condition to receive the water. The juice from all the mills is mixed and is pumped to the defecators for purification.

In **double saturation**, all the water is applied to the bagasse from the second mill. The thin juice from the third mill is strained and is used to saturate the bagasse from the first mill. The mixed juices from the first and second mills are pumped to the defecators.

36. Diffusion Process.—As the **diffusion process** is used exclusively in beet-sugar manufacture, and to only a very limited extent in cane-sugar factories, it will be described fully in the Sections devoted to the beet-sugar industry. A brief outline of the process as applied to cane, however, will be given here.

The process as used in American factories in extracting the sugar from cane is usually maceration rather than diffusion. The cane is reduced to fine shreds, or match-like fragments, and is then introduced into a series of cylindrical iron vessels, where it is systematically soaked in water. The work is so conducted that the first of the series of iron vessels, or diffusers, contains exhausted cane and the last, fresh cane and concentrated juice. The exhausted cane is rejected and juice from the last diffuser is sent to the defecators from time to time, for purification. When a diffuser is empty the succeeding one becomes first in the series, and a freshly filled vessel becomes the last. The liquid near the end of the series of diffusers is heated to a temperature of about 85° C.

Owing to the scarcity of water and the high price of fuel on most plantations and to the difficulty of disposing of the residue, or exhausted chips, this process is little used in cane factories. From the point of view of the extraction of the sugar, however, it is an ideal process.

37. Diffusion of Bagasse.—A diffusion process, or, correctly speaking, a maceration process, for treating bagasse in a diffusion battery has been patented by Naudet. As this process combines with it the purification of the juice, it will be described under that heading in Art. 54. The maceration of the bagasse in a diffusion battery has been frequently applied.

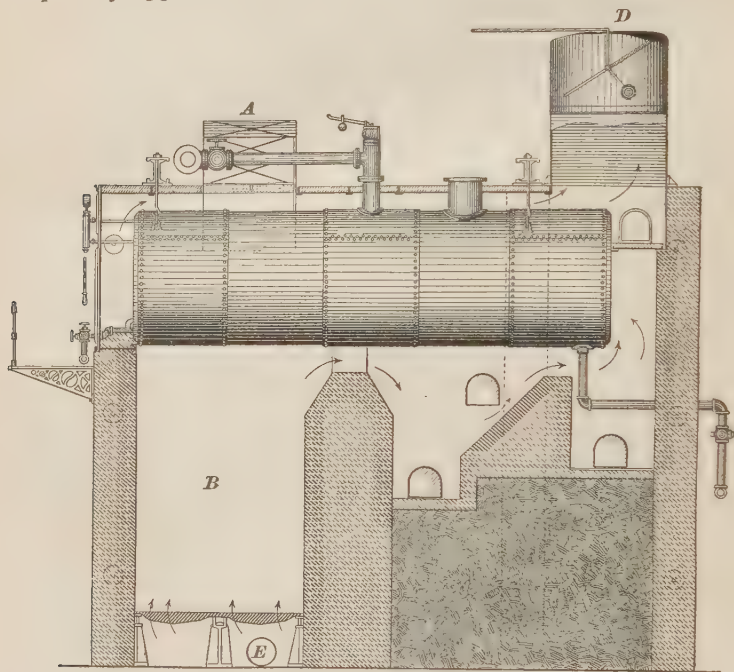


FIG. 3

38. Disposal of Bagasse.—The cane bagasse, under good milling conditions, contains from 50 to 53 per cent. or less of water, and from 6 to 8 per cent. of sucrose. The bagasse makes excellent fuel, but it must be burned in a special type of furnace. Few, if any, factories utilize the bulk of their bagasse for any other purpose.

The furnace used varies somewhat with the type of steam boiler employed. A *Fiske burner*, or *furnace*, Figs. 3 and 4,

with two multitubular boilers, illustrates one of the usual appliances for utilizing the bagasse for producing steam.

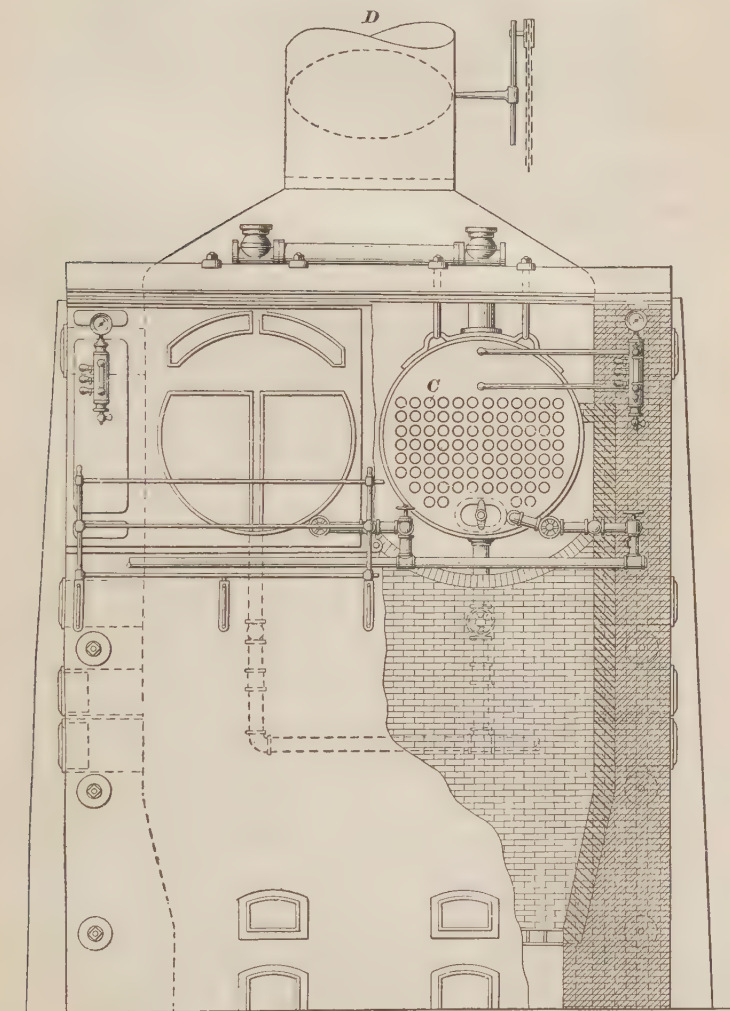


FIG. 4

On leaving the last mill, the bagasse is elevated to the automatic feeder *A*, the balanced door of which opens when

the fuel falls on it and immediately closes after the fuel passes. This automatic feeding of the bagasse is very important, as it is necessary, as far as possible, to avoid the admission of air to the furnace above the grate bars. From the feeder, the bagasse falls between the boiler shells directly on the fire in a large combustion chamber. Air is forced under the grate bars by a pressure blower through the opening *E*. Owing to the nature of the fuel and the large quantity of moisture it contains, the combustion chamber *B* is very large and a very high temperature must be maintained in it. This temperature attained in the combustion chamber is very often $1,300^{\circ}\text{C}$. and frequently reaches $1,400^{\circ}\text{C}$. On account of this high temperature, it is necessary that the gases have a long distance to travel in contact with the shell of the boiler before entering the flues. With the Fiske type of furnace, the multitubular boilers for a large plant should be about 22 feet long. The heated gases pass under the shell of the boiler, then through the tubes *c*, Fig. 4, to the front end, and then over the boiler to the chimney *d*. Every part of the boiler is exposed to the heat. Owing to the intense heat, a part of the ash fuses to a slag. This slag adheres to the firebrick lining of the furnace and must be removed at intervals.

When water-tube boilers are used, the furnace is usually placed between two boilers.

In the Cook type of furnace, which is also used extensively, the bagasse is burned on a hearth instead of on grate bars, and the air is forced into the burning material through tuyères.

39. Where the installation of machinery in the factory is economical and the cane rich in woody fiber and sucrose, the bagasse furnishes nearly sufficient fuel for the manufacture of the sugar. It is largely supplemented in Louisiana by fuel oil and in the tropics by wood. In modern methods of manufacture, in which only high grades of sugar are produced, more fuel is required than in the production of first sugar and molasses sugars.

The nominal steam-boiler capacity required by a factory varies between very wide limits. It depends on the richness of the cane in sugar, on the grade of sugar made, and on the mechanical equipment and methods of manufacture. A capacity of 700 to 800 nominal horsepower is usually sufficient for a factory that grinds 500 tons of cane in 24 hours.

It has been proposed to use the bagasse for the manufacture of paper stock, and machinery has been installed for that purpose. The results, however, have not been commercially satisfactory.

Small quantities of finely ground bagasse are used in the manufacture of a cattle food called "molascuit," in which the bagasse serves as an absorbent for molasses.

PURIFICATION OF JUICE

40. Juice Strainer.—As it flows from the mill rolls, the juice carries with it a large quantity of fine bagasse, which is termed "trash" or "cushcush." The latter word is used only in the English factories. In well-equipped sugar houses, the juice flows from the pans of the mill on a brass screen that has from 400 to 600 round perforations per square inch. The holes of this screen are prevented from clogging, and the trash is elevated and deposited in front of the second mill by a return-flight, link-belt conveyer, or drag. The flights, or scrapers, are edged with rubber belting.

After being strained, the juice is pumped either to the defecators or to the liming tanks, according to the method of further purification that is used.

41. Defecators and Clarifiers.—The treatment of the juice after being strained depends on whether raw sugar or white sugar is to be made. As comparatively few factories make the latter, the raw-sugar methods will be described first, and later the modifications necessary in making white sugar.

Defecators, as shown in Fig. 5, are usually round iron vessels with double hemispherical bottoms, the inner bottom

being of copper. In heating the juice, steam is admitted to the space between the bottoms. This type of defecator is used almost exclusively in tropical sugar factories.

The American factories use a rectangular iron tank fitted with steam coils for the defecation, and usually term it a **clarifier**, and the process the *clarification*. As will be shown later, the defecation is a distinct process from the clarification. The tropical factories often dispense with the latter process, and those of Louisiana usually accomplish both in the rectangular defecator, or clarifier. In any case, several of these tanks are always used, their number and capacity varying with the size of the factory. As the tanks

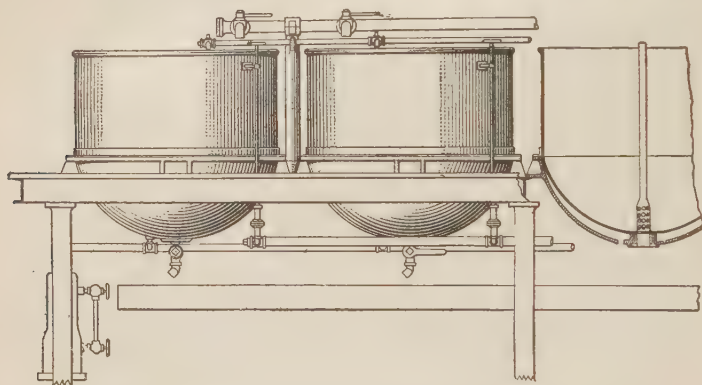


FIG. 5

are used in regular rotation, a sufficient number is installed to allow time for the separation of the impurities and the decantation of the clean juice. Large factories generally employ more than one series of tanks. The rectangular tanks, however, may be readily constructed of sufficient capacity for very large establishments without duplicating the series. Each defecator is provided with an inlet pipe for raw juice; draw-off pipes, cocks, and gutters for clean juice and scums; and a removable perforated plug, to prevent the scum from mixing with the clear juice during the decantation. A steam inlet and an outlet for the water of condensation are also provided. The clarifier differs from the defecator

in having a trough to receive scum that may be brushed from the surface of the defecated juice.

42. A method of defecation and clarification that is extensively used is known as *Deming's system*. This method employs closed steam digesters and closed or open settling tanks of special form. The prominent features of this process are the heating of the juice under pressure to a temperature above its boiling point in the open air, and its constant action, the ordinary process being an intermittent one.

43. A type of clarifier, much used by English factories, and of very efficient construction, is the **eliminator**. This apparatus consists of a rectangular tank in which the juice is heated by a steam manifold having brass or copper tubes. The steam is admitted to the rear end of the manifold, and in boiling the liquor, it throws the scum to the front of the tank. Here, the scum overflows into two scum compartments, which extend parallel with one another across the front of the clarifier. The inner compartment has a row of small holes in the partition wall, through which juice that separates from the scum may flow back into the tank. The heavy particles of scum are either carried by the boiling liquor or lifted by the attendant into the outside compartment, whence they flow through a pipe to receiving tanks, preparatory to filtration.

In the ordinary clarifier largely used in Louisiana, after the heavy scum is removed, the juice is boiled and the scum that rises is brushed into a trough.

44. Ordinary Process of Defecation and Clarification.—The defecation process, as the name indicates, consists in separating the impurities of the cane juice that are coagulable by heat and also those precipitable by lime. Of the impurities, the nitrogenous bodies, including the albuminoids, are partly removed by coagulation, and the pectin, fat, and wax, and the mineral and organic acids are precipitated in part by the lime or are removed mechanically by the precipitates.

In using the ordinary defecators, the juice is drawn into the tank until the copper bottom is covered. When the tank is filled, sufficient lime, in the form of a milk, is added to render the entire charge slightly alkaline, and steam is then admitted to the heating chamber between the double bottoms. The admission of steam is so regulated that when the juice reaches a point about 6 inches below the rim of the tank, its temperature is only a few degrees below its boiling point. The stream of juice is now turned into the next defecator of the series. The heating of the juice in the first tank is continued until the gases liberated just break the heavy surface scum, or "blanket." This is technically termed the *cracking point*. At this stage, the steam is shut off from the defecator and the juice is left at rest, so that the heavy precipitates will sink to the bottom of the tank and the flocculent matter will rise to the surface of the liquid.

If the proper proportion of lime has been used, the precipitates will form a compact mud on the copper bottom, the juice will soon separate in part from the blanket, leaving the latter firm and porous, and the juice between the mud and the blanket will be bright and sparkling. The impurities will usually settle well even if a considerable excess of lime has been used; but insufficient lime, on the contrary, produces a turbid, or cloudy, juice. In raw-sugar manufacture, the lime should be added to slight alkalinity; but in making white sugar, the limed juice should be faintly acid to litmus paper. The precipitates and flocculent matter separate well from both alkaline and slightly acid juice. The sugar maker usually judges the quantity of lime to use by the indications already described and by the appearance of the scum and the rate at which the floccules move in a test sample of the defecated juice. By these indications, he is guided in liming the next and subsequent defecators of juice.

45. The quantity of lime to be used in making raw sugar may be estimated by the following simple chemical test: To a small portion of defecated juice that has been filtered through paper, add a solution of saccharate of lime,

a few drops at a time, noting whether or not a precipitate forms. A glass test tube should be used in this experiment. If a precipitate appears, the juice has not received sufficient lime; if no precipitate forms, either too much or just enough lime has been used in the defecation. If a precipitate forms or does not form, the liming of a subsequent defecator of juice should be changed accordingly, and the test should be repeated. The addition of lime should be continued as long as a precipitate appears.

46. After allowing sufficient time for the separation of the impurities, the draw-off cock at the bottom of the defecator is opened and the cloudy juice is run into a suitable gutter leading to the scum tanks. When the clear juice follows, it is run into a trough leading to the clarifiers or storage tanks, and, finally, when the juice becomes turbid, the central plug is removed and the mud is diverted into the scum canal.

Where the combined defecators and clarifiers are used, the procedure is the same as already described, except that the blanket scum is removed with a paddle, called a *brush*, and the juice is boiled and skimmed, that is, clarified. After this treatment, the juice is allowed to remain for some time in the clarifier, which is used in lieu of a settling tank, or subsider, and is then decanted into the storage tanks.

47. In the manufacture of raw sugar, the juice in certain factories is limed in excess and then passed through a sulphur box or other sulphuring device, where sufficient lime is precipitated by the sulphurous acid to reduce the alkalinity of the juice to that in the ordinary process. It is claimed that this process yields very pure, free-boiling juices, and that the molasses has less viscosity than would otherwise be the case.

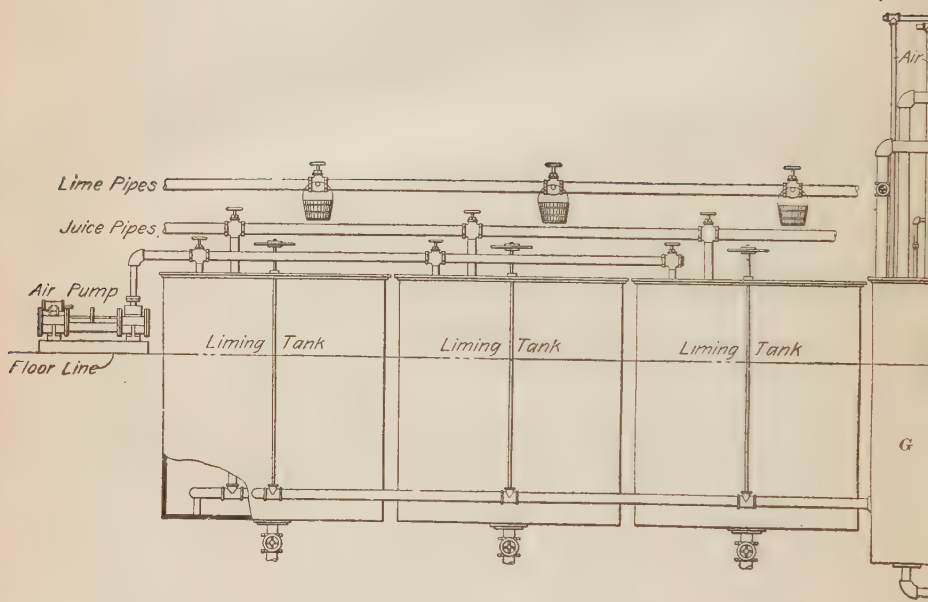
In producing white sugars, the cold juice, just as it flows from the strainer, is saturated with sulphurous-acid gas, is then limed to nearly neutrality to litmus paper, and the defecation is conducted as already described. This process is also used to some extent in making Louisiana raw sugars, but

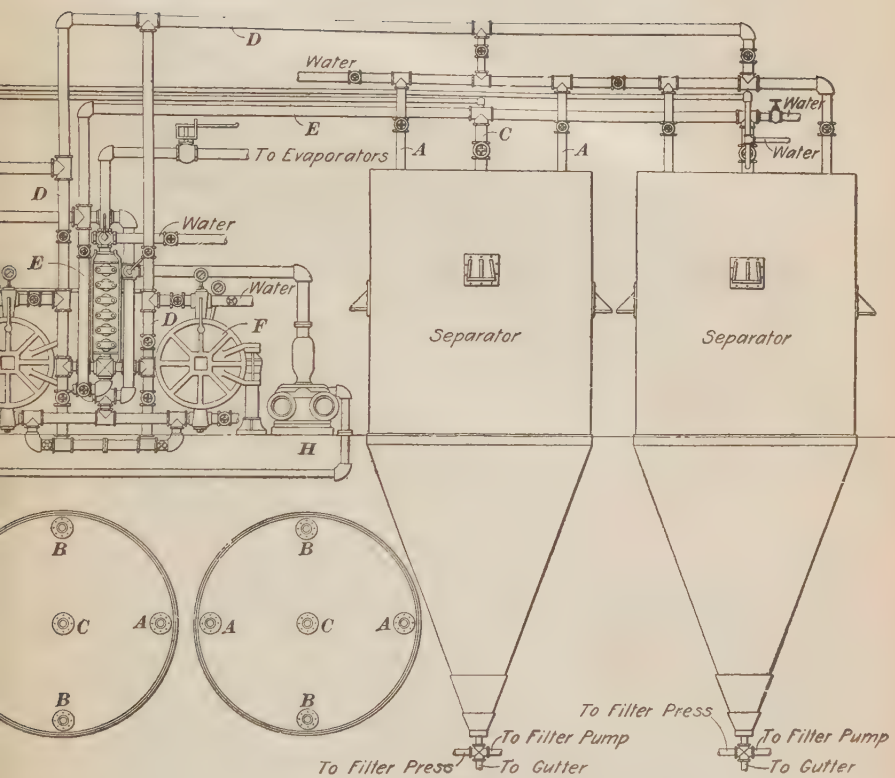
where so employed in the tropical factories, lime is added to the juice to alkalinity. Phosphoric acid is occasionally used in the manufacture of white sugar. The juice is limed in the defecation to slight alkalinity to litmus paper. After the removal of the scum, by decantation or otherwise, sufficient phosphoric acid is added to neutralize the excess of lime, and the juice is then brushed or clarified.

In still another modification of the defecation process, the juice, after liming, is heated to near its boiling point in a tubular heater. It is then run into an ordinary defecator, where the heating is continued until it boils. In this process the separated impurities settle to the bottom of the defecator, instead of remaining partly at the surface. This behavior of the precipitates is due to the expulsion of the gases from the juice during the boiling.

48. Deming's System of Defecation and Clarification.—The system of defecation and clarification known as **Deming's method**, is the only radical change that has been made and extensively applied in the purification of cane juice since the early days of the cane industry. In this method, the juice is limed in the cold in a series of tanks shown at the left in Fig. 6. At the right of the group of liming tanks and connected with each by suitable pipes and valves is the pump tank *G*, which is deeper than the other tanks and into which the latter may be drained. The lime, in the form of a milk, is added to the juice from time to time, as the liming tank is filling. Agitators, not shown, prevent the lime and precipitates from settling to the bottom of the tank. The air pump shown in the figure is used in many installations to force air through perforated pipes into the juice and thus agitate the latter. The quantity of lime to be used is ascertained from an examination of the clarified juice, as in the ordinary process.

In addition to the liming tanks, a digester, an absorber, and a series of settling tanks are required. The digester is often installed in duplicate so as to facilitate the cleaning of its tubes or to increase its heating surface.





The digester is a cylindrical iron vessel fitted with copper heating tubes. In the figure, the digesters in duplicate are shown at *F*. The absorber is located between the digesters and is composed of a system of wrought-iron pipes, in each of which is a copper tube. The iron and copper pipes have suitable head-pieces and return bends for the circulation of the juice. In operating the apparatus, the juice is pumped by means of the juice pump *H* from the liming tanks, around the copper tubes of the absorber, and then through the tubes of the digester, where it is heated by steam to a temperature of 112° C. or even higher. From the digester the juice returns to the absorber, passing through its tubes. The function of the absorber is to reduce the temperature of the juice to below its boiling point in the open air, thus preventing it from flashing into steam when the pressure is reduced. In this way, the absorber tends toward economy in fuel. It is usually sufficient to pass about three-fourths the total volume of the cold juice through the absorber, so as to reduce the temperature of the clarified juice to below its boiling point. The apparatus should be operated at a minimum juice pressure of 25 pounds, as indicated by a gauge. As the heating surfaces foul, the pressure rises, but it should not be allowed to exceed 50 pounds.

49. From the absorber, the juice flows into conical-bottomed settling tanks, or separators, shown at the right in Fig. 6. The settling tanks shown here illustrate a recent improvement in this apparatus. In this case, the tanks are closed, and they are termed "separators" by Deming. Inside of each tank is a chamber shaped like the frustum of a cone, but open at both top and bottom and with the large opening below. The settling tanks are arranged in series. The clarified juice passes into the outer chamber of the first, or primary, tank, flows downwards to the inner chamber, and then upwards and through the overflow pipe to the secondary settling tank. As the surface area of the juice in each chamber is large in proportion to the quantity of juice flowing through it, the current is slow. The descending

juice carries the precipitates with it, and as it rises in the inner chamber to the overflow, it leaves a large part of them in the conical bottom. The flow of juice through all parts of the apparatus is constant while the mills are in operation. From time to time, a cock at the lower part of the settling tank is opened and the mud is drawn off into the scum tanks.

On leaving the digesters, the juice, under pressure, passes through the pipes *D* and *A* into the separators and flows from these to the pipe absorber through *C* and *E*. From the absorber it passes to the charge tanks of the multiple-effect evaporator. The temperature of the juice is reduced to about 82° C. in the absorber.

50. Carbonatation Process.—In treating cane juice, a modification of the carbonatation process used in beet-sugar factories is applied extensively in Java, but in other places it is used only to a very limited extent. This process gives excellent results from a sugar-making point of view, and its limited application is due only to the high working expense.

As this process is the only one used in beet-sugar manufacture, it will be described fully later. Only the modifications necessary in its application to cane sugar will now be considered. The carbonatation may be done by either the *single* or the *double method*.

51. Single Carbonatation.—In the **single-carbonatation method**, the juice is rendered strongly alkaline with a quantity of milk of lime equivalent to about 1.25 per cent. of its weight of quicklime. A smaller quantity often suffices. The lime is then saturated with carbonic acid. During the carbonatation, the temperature of the juice is maintained at or below 60° C., but as soon as the lime has been completely precipitated, it is quickly raised to above 90° C. and the juice is then filtered.

52. Double Carbonatation.—In the **double-carbonatation process**, the lime is added in two portions. After the first liming, carbonic acid is forced into the juice until the alkalinity of the latter is approximately equivalent to

1 gram of quicklime per liter, a temperature not exceeding 60° C. being maintained throughout the operation. After freeing the juice from the precipitate by filtration, it is again limed, but only a very small proportion of the reagent is used. After the second liming, the juice is again carbonated and further treated, as in the single-carbonatation process.

The temperature is kept at 60° C. or lower while the juice is strongly alkaline. This is done to prevent the decomposition of the invert sugar and the consequent formation of highly colored products. The presence of an excess of lime at the end of the first carbonatation, in the second process, is to prevent a part of the precipitate from being redissolved. The double carbonatation yields purer juices than the single process.

53. In the manufacture of white sugar by the carbonatation process, after the nearly complete precipitation of the lime, the juice is "sulphured," that is, treated with sulphurous acid, until its reaction is faintly acid to litmus.

Carbonated juices, as well as those sulphured after a single or double carbonatation, may be readily filtered through filter presses. For the filtration of juices clarified by the ordinary processes, mechanical filters that work under low pressures are necessary. Mechanical filters will be described later. The clarified cane juice is not usually filtered.

54. Naudet Process of Extracting and Purifying Juice.—In the Naudet process, which is controlled by the Harvey Engineering Company, of Glasgow, the extraction of juice from the bagasse and the purification of juice from both the cane and the bagasse are carried on simultaneously.

The cane is first crushed or shredded and passed through the first mill, as previously described in connection with the milling processes. The bagasse from this mill is extracted in a diffusion battery of eight cells, first with very hot juice and then with water. The diffusion battery is similar to the battery that will be shown later, in *Manufacture of Sugar*, Part 3, though the cells may be arranged in a circle or straight line, as may be convenient, and the circulation of

the juice in the last vessel is accomplished by means of a pump.

55. In conducting the Naudet process, diffuser No. 1 is filled with bagasse; then water is circulated through it and the heater, until the thin juice formed acquires a temperature of from 200° to 220° F., when it is forced into a mixing tank. In the meantime, diffuser No. 2 has been filled with bagasse and the thin juice from the mixing tank is circulated through it, as in the case of the previous vessel. While heating the juice and exhausting the bagasse in No. 2, water is passed through No. 1 and into the mixing tank, so as to supply any deficiency there may be in the quantity of thin juice and to wash the partly exhausted bagasse. These operations are repeated with fresh bagasse in diffuser No. 3, the water passing through Nos. 1 and 2 to the mixing tank, and so on until six diffusers are in operation. The object of these manipulations is to extract the sugar from the bagasse.

Assuming that a measuring tank has been filled with normal juice from the mill and that diffuser No. 7 has been filled with bagasse, the normal juice is run into No. 7 and half fills the diffuser. The juice in the mixing tank is limed and is run into No. 7 until the cell is completely filled; and it is then circulated through this diffuser by the pump until its temperature is raised to about 200° to 220° F. During this operation, the juice is defecated and the precipitates are filtered out of it by the circulation through the bagasse. As soon as the desired temperature has been reached, a measured quantity of juice is drawn from diffuser No. 7 and is sent directly to the evaporators to be concentrated to sirup. The circulation of the juice is forced by compressed air or by water pressure in drawing the juice. When the measured quantity has been drawn, the current is diverted into the mixing tank, to be used in diffuser No. 8. While No. 8 is filling with bagasse, No. 1 is cut out of circulation and opened, and the exhausted bagasse that it contains is discharged on a carrier and conducted to a second mill, where it is pressed preparatory to use as fuel. The water

expressed from the bagasse contains only a trace of sugar and is run to waste. As in the diffusion process, each time that a charge of juice is drawn, the first diffuser of the series is cut out and the exhausted bagasse is removed.

56. Remarks on Defecation and Clarification Processes.—Many processes have been devised for the purification of cane juice, but only those that have been described are in use. In very small factories, an open fire is often used instead of steam in the defecation, clarification, and concentration of the juice, all of which are accomplished in open kettles.

The methods that have been used and discarded include, among others, the following: processes employing clay or alum to increase the rate of the settling of the precipitates; bisulphite of lime, for bleaching and precipitation; albumen, to promote flocculation; also, electrolytic processes and several processes employing bisulphites and hyposulphurous acid.

57. The clarified juice is far from being a solution of sugar and water. Many of the impurities cannot be removed from the juice except by processes that are not commercially profitable. The impurities that remain in the juice impede the crystallization of the sugar and are the cause of the final molasses by-product. The impurities also separate from the liquors to some extent during the concentration and thus foul the heating surfaces of the evaporators. The heating surfaces of the defecators and clarifiers also become foul, the scale formed consisting largely of lime compounds and silica. As the scale is a poor conductor of heat, the surfaces must be cleaned frequently.

The copper surfaces of the double-bottomed defecators may be cleaned by scouring. This and other types of defecators are more readily cleaned, without delaying the manufacture, by covering the copper surfaces with water that is strongly acidulated with muriatic acid. The acidulated water should be boiled for a few minutes, and then run into the sewer. From 4 to 6 pounds of the commercial acid is required to clean a 700-gallon, double-bottomed defecator.

58. Chemical Reagents Used in Purifying the Juice.—*Lime* is used in all the processes now employed in purifying cane juice. The following methods are used in preparing the lime:

1. Quicklime is ground to an impalpable powder, sifted, and stored in covered casks, or barrels. This lime should be slaked in water immediately before using it.

2. Quicklime is heaped on a floor, and then sprinkled with water so as to slake it and produce a dry powder. For use, this powder should be sifted and reduced to a milk, as required, with raw juice.

3. Milk of lime, reduced to 18° Baumé, is circulated through pipes by a special pump and returned to the pump tanks. This circulation of the liquid keeps the lime in suspension. The milk is drawn off where required, from branch pipes, and a measured quantity is used in liming the juice.

4. A quantity of quicklime is slaked and reduced to a milk, and the liquid is strained into a box or a tank. After allowing sufficient time for the lime to settle, the supernatant lime water is drawn off, leaving a thick lime paste. Weighed portions of the paste are diluted with juice, as required, for use in the defecation.

When dry lime is used, whether lime powder or dry slaked lime, the charge for each defecator should be weighed and not measured.

59. Sulphurous acid is always used in the gaseous state in cane-sugar factories. This acid is produced by the combustion of sulphur in the air, in a stove designed for that purpose. The stoves employed are of two types, namely, *closed*, into which air is forced by a pump; and *open*, into which air is drawn by natural draft, by the juice pump, or by a steam ejector. The first form is seldom used in the cane-sugar industry, being employed almost exclusively in beet-sugar manufacture.

60. The open type of stove is of cast iron and is usually shaped like an inverted letter **U**, in cross-section. A large cast-iron pipe leads from the dome of the stove to the saturating

device. Both the stove and the pipe should be water-jacketed. The sulphur is burned in a pan at the bottom of the stove, and the quantity of air admitted is regulated by a damper. The saturating apparatus is often a pump, which draws sulphurous-acid gas from the stove and juice from the mill tank and then mixes and delivers the two at the defecators.

A very common saturation device consists of a tall wooden box or column fitted with either slanting or perforated shelves. The mill pump delivers the juice into the top compartment of the box, from which it falls in a shower or series of cascades to the bottom of the column and is carried by a pipe to the defecators. At the same time a current of sulphur fumes is drawn into the lower part of the box and travels upwards, meeting the shower of juice and being largely absorbed by it. A flue at the top of the box carries off the surplus fumes and air. This flue may be made of either wood or lead. If the natural draft is not sufficient, a small steam nozzle directed upwards in the flue will produce the desired result. The nozzle should be of lead hardened with antimony.

61. Sulphurous acid bleaches the coloring matters of the juice and in doing so is partly oxidized to sulphuric acid. In common with other mineral acids, it has the property of coagulating albuminoids. If juice acidulated with this acid is heated or is not promptly neutralized, a loss of sucrose results through inversion. This reagent has almost entirely superseded animal charcoal in the production of white sugar in cane- and beet-sugar factories.

FILTRATION OF JUICE AND SCUMS

62. Filtration of Juice.—Filter presses and filters will be described later in connection with beet sugar and sugar refining. Clarified juice is difficult to filter, except when obtained in connection with the diffusion process or by carbonatation. Clarified diffusion juice may be strained

through bag filters, such as are used in refineries. Carbonation juices filter readily through all types of filters. Juice defecated and clarified by the ordinary process filters with great difficulty through cloth in filter presses. The minute particles of almost invisible flocculent matter suspended in the juice soon coat the cloths and prevent filtration. The defecated juice, however, may be filtered with very low pressure through cotton cloth or sand in the so-called mechanical filters. In the cloth filters of this type, the mud collects on the outside of the bag and remains soft and pervious, falling from the cloth from time to time as it accumulates.

Fine bagasse is used to a limited extent as a filtering medium. The bagasse soon clogs with the flocculent matter of the juice and is then returned to the mills to be reground with other bagasse.

63. Filter Pressing of Scums and Precipitates.

The scums and precipitates from the defecation and clarification are collected in blow-up tanks preparatory to filtration with presses. These tanks are fitted with perforated pipes, for blowing steam into the scums, and also with cocks at different levels, for decanting the clear juice that separates from the material after heating it.

In the manufacture of either raw or white sugar, it is often advisable to lime the scums to alkalinity, and then heat them with the blow-up pipes to the boiling point. After heating, the contents of the tanks should be left at rest, for the subsidence of the precipitates and the subsequent decantation of the clear juice. This treatment promotes the filtration and reduces the loss of sugar in the press cake. The fine flocculent matter in the thin scums sometimes forms an almost impervious coating on the filter cloths and greatly reduces the capacity of the presses. In the material prepared by liming, heating, and decantation, the porous blanket scums and the heavy precipitates form an excellent filtering medium, and no trouble is experienced in the filtration. Some factories, however, do not prepare the scums as

just described, but in many instances this method has given satisfactory results.

The scums may be forced into the press either by a *monte jus*, that is, by direct steam or air pressure, or by a pump. The latter, however, is preferable. The pressure in the press should not exceed 60 pounds, while a much lower pressure is usually sufficient. In factories having enough tank and evaporator capacity, a saving of sugar is effected by diluting the scums with water after draining off as much juice as possible.

64. Filter presses are usually arranged so that the washing of the residue, or press cake, may be done systematically by passing water through the cake and thus driving out the juice it contains. This method demands very large filter-press capacity, however, and is seldom used except in beet-sugar factories.

The filter-press work is frequently seriously impeded by mixing the precipitate or mud that collects in the sirup tanks with the scums. It is preferable to mix this precipitate with the raw juice before defecation. Where clarification follows the defecation and the process is carried on in separate tanks or in combined defecators and clarifiers, the mud washed from the sirup tanks may be returned to the clarifiers. Care, however, should be taken that this material is largely diluted with juice, otherwise difficulty may be encountered in separating the mud from the clear juice in the subsidiers. Should the mud tend to rise to the surface of the juice during settling, the surface of the juice should be liberally sprayed with cold water, which, in falling to the bottom of the tank, usually carries the flocculent matter with it.

With the Deming process of clarification, the sirup-tank washings, or "tank bottoms," as such material is usually termed, should be gradually run into the pump tank to be reclarified. Where it is impracticable to treat the tank bottoms as recommended, they should be run into a separate blow-up tank and then heated to the boiling point. After

settling, the clear liquor should be decanted. Liberal quantities of water should then be used in diluting this material, preparatory to steaming and filtering it. The juice from the filter presses is usually pumped directly to the charging tanks of the evaporators. Where practicable, the juice should first be reclarified, because it is often cloudy, owing to a defective or badly adjusted cloth. Each filter plate of a press is provided with a juice cock for disconnecting it in the event of a cloth breaking. When a cloth is badly adjusted, the press must usually be opened and dressed with clean cloths. This, however, involves loss of time and additional labor for washing the cloths; therefore, when possible, it is preferable to let the press fill slowly under very low pressure. While this may result for a time in a cloudy filtrate, the opening at the defective cloth often fills with the precipitate, permitting the filtration to proceed as usual.

The filter cloths may be used several times without being washed, provided the press is completely filled with well-pressed cake. The latter should leave the cloth freely, soiling it but little. The workman can tell when to open a press by noting whether the juice dripping from the cocks has almost completely ceased, or by closing the inlet valve and drawing the mud from the channel and then noting its volume. If the volume of this mud is greater than that of the channel, it is evident that the press is not full of hard cake. The filter cloths are washed and dried by means of the appliances used in large laundries.

MANUFACTURE OF SUGAR

(PART 2)

MANUFACTURE OF CANE SUGAR

(Continued)

CONCENTRATION OF JUICE TO A SIRUP

1. The juice having been freed from impurities, so far as the usual methods of manufacture permit, it is concentrated to a sirup, preparatory to a final concentration with crystallization of the sugar, in a vacuum pan.

The evaporation of the juice was formerly conducted in open vessels that were worked in batteries, or trains. Apparatus of this class is now used only in very small factories and especially in those located in remote tropical regions, where local market and transportation conditions render work on a small scale profitable.

2. Evaporation in Open Kettles and Steam Trains.

A battery of open kettles is illustrated in plan and in section in Fig. 1. The kettles *c, d, e, f* are arranged over a furnace and flue, each at a level somewhat lower than the one preceding it. The kettles *a, b* near the chimney are used as clarifiers, from which the juice is drawn into kettle *c*. The entire series of vessels is heated by means of the furnace *g*, the fire being directly under kettle *f*, while the heat for the other kettles passes through the arches *h*. The kettle *f* is thus in contact with the highest temperature.

In operating the train, the kettles are filled with juice, which is then heated to boiling. As the water evaporates, the juice is ladled from kettle to kettle, to compensate for its decreasing volume, and at the same time the scum that rises to the surface is removed with a skimmer. The kettle *f* is termed the *strike pan*. The sirup is concentrated in this kettle to the point of crystallization and is then ladled into

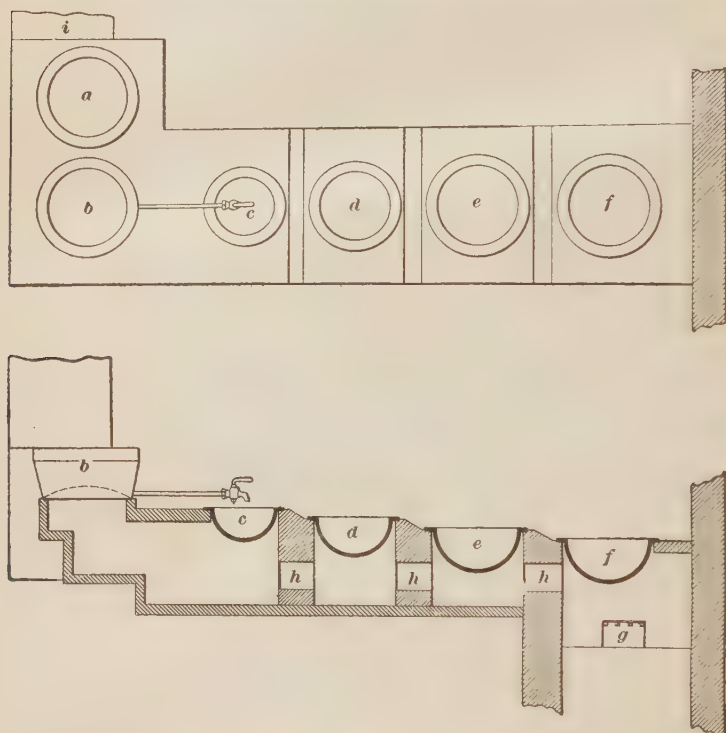


FIG. 1

wooden coolers, where it soon forms a mass of crystals and molasses. In many tropical countries, notably in parts of Mexico and Central America, the evaporation is carried to such a point that the dense sirup solidifies on cooling in suitable wooden molds, forming a very dry concrete that may be wrapped in leaves and shipped in this form for immediate consumption.

3. The steam train differs from the kettles in that steam is used for heating instead of an open fire. The pans of the train are sometimes connected with one another, so that the liquor may flow from vessel to vessel. There are several modifications of the steam train. These include evaporators consisting of a single vessel, into which a constant stream of juice flows at one end and heavy sirup is drawn off at the other.

4. **Multiple-Effect Evaporation.**--All large factories concentrate the liquors in vacuo in **multiple effect**. With multiple-effect evaporators, as the name indicates, the heat applied in boiling the liquor is not lost after one application, but, in the form of the steam generated in this work, is made to evaporate the liquid in a second vessel, and so on through several applications. In order to accomplish this multiple application of the heat, the vessels, or pans, are closed and the air is partly exhausted from them, the vacuum increasing from pan to pan, thus reducing the temperature at which the liquid boils. The steam generated in boiling the liquid in the first vessel passes through a suitable pipe to the heating surface of the second pan, where it is condensed in evaporating the liquid in this vessel, and thus produces a partial vacuum in the first pan of the system. A similar utilization of the vapor generated in the second vessel is applied in the third, and a partial vacuum is produced in the second vessel. The last vessel of the series is connected, through a condenser, with a vacuum pump, which produces a very high vacuum in this pan. The water of condensation is led off from the first pan through a trap, and is removed from the others by one or more pumps. This water is generally utilized in feeding the boilers, so besides furnishing a very pure water for this purpose, it supplies the boilers with water that is partly heated.

It is evident that the economy of fuel with multiple-effect evaporation is very large. Roughly speaking, assuming that fuel equivalent to 1 pound of coal burned under a boiler will evaporate $7\frac{1}{2}$ pounds of water, in a multiple effect composed

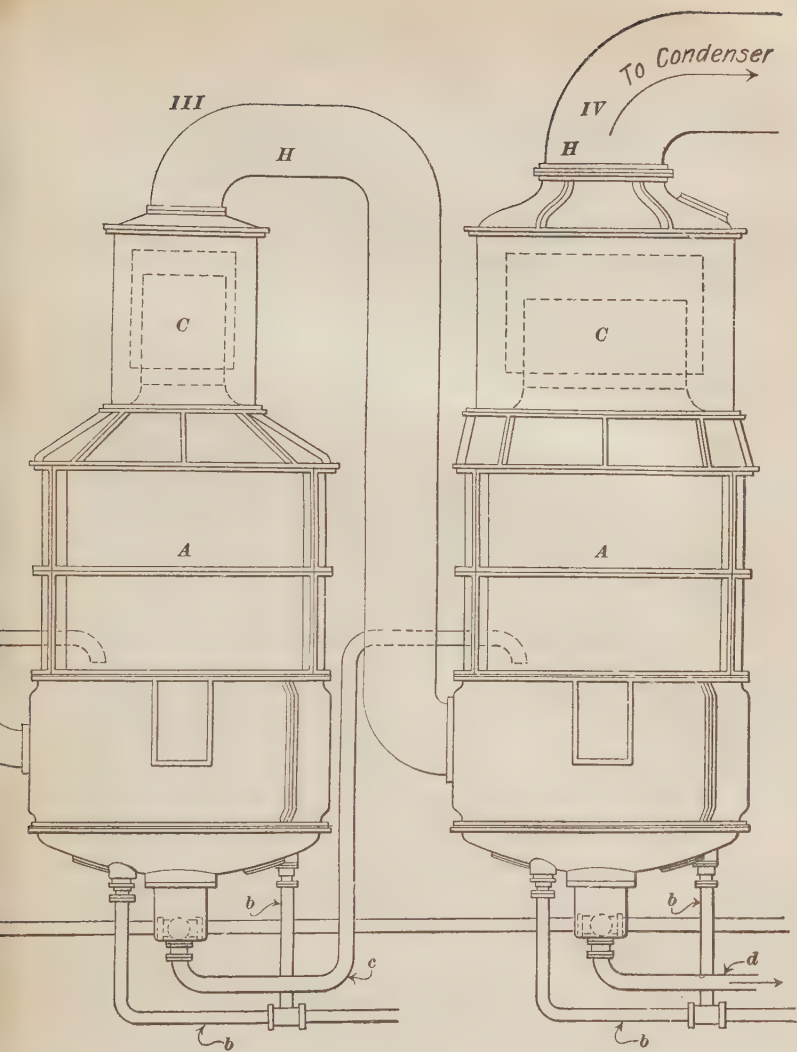
of two vessels, it will evaporate 15 pounds of water, and in a system of three pans, $22\frac{1}{2}$ pounds. These figures are only rough approximations. Exhaust steam from the various pumps and engines of the factory is used for multiple-effect evaporation.

5. Kinds of Multiple Effects.—Multiple effects are usually composed of two, three, or four vessels, and are termed, respectively, *double*, *triple*, or *quadruple effects*. At the present time, double effects are seldom used. The triple effect is usually installed in modern factories, though many large establishments use quadruple effects. A larger number of vessels than four in a series is rarely employed.

There are many types of multiple effects in use in the sugar and other industries, but the principle on which these are based is the same for all. The form of multiple effect usually employed in the cane-sugar industry is shown in Fig. 2, and is called the **standard type of evaporator**. Since it is very important that the construction and manipulations of a multiple effect should be understood, this apparatus will be described in detail.

6. Quadruple-Effect Evaporator.—The evaporator shown in Fig. 2 is based on the drawings of a large quadruple effect, having 12,500 square feet of heating surface, that was designed and constructed by Samuel Vickess, M. E., New York, for a factory very recently built in Porto Rico. Only the parts of the apparatus essential to an understanding of its construction and manipulation are shown in the illustration. The dimensions are those of the Porto Rico quadruple effect, and are given to illustrate the variations in the construction of certain parts of the vessels that were necessary on account of the differences in vacuum.

Referring to Fig. 2, *A, A, A, A* are cylindrical cast-iron vessels, or pans, each 9 feet 3 inches in diameter. At *D* of each vessel there is a drum, or belt, fitted with a large number of brass tubes and a large iron tube *E*. Each brass tube is 5 feet $1\frac{1}{2}$ inches long by $1\frac{1}{2}$ inches in diameter, and the tube *E* is 3 feet in diameter. The drum *D* is called the





calandria, and the tube *E*, the *down-take*. A section of the vessel through the *calandria* shows the arrangement of brass heating tubes *D*, and the down-take *E*.

7. When the apparatus is in operation, fine particles of juice are projected from the boiling liquor and are carried upwards with the vapors. This is termed *entrainment*. To avoid the loss of this juice, save-alls are located in the vapor pipes at *B*, *C*, *C*, *C*. The vapors follow the path in the save-all indicated by the arrows. As shown at *B*, a hood with serrations on its lower edges fits over the neck of the vessel. The particles of liquid entrained with the vapors strike against the walls of the hood and are held there until they drip to its lower edge. Here, the serrations gather them into large drops, which fall into a suitable canal and are led back into the vessel through the pipes *f*. These pipes are curved at the lower ends, so as to trap a portion of the liquid and thus prevent the vapors from passing through them.

The vessels are connected with one another by pipes *H*, which lead from the vapor space of one to the *calandria* of the next, with the exception of the vapor pipe of the last pan, which is connected, through a condenser, with a powerful vacuum pump. The types of vacuum pumps will be described later.

8. Since, as just explained, the first vessel of a quadruple effect is usually operated under low pressure and the others with a vacuum, which increases from pan to pan, the volumes of the vapors from equal quantities of boiling liquor also increase from pan to pan, being greater the higher the vacuum. Provision for this increase in volume is made by increasing the size of the save-alls *C* and the vapor pipes *H*. The diameters of the necks of the vessels at the save-alls *B*, *C* in the Porto Rico apparatus are, respectively, 2 feet, 2 feet 2 inches, 2 feet 9 inches, and 5 feet 6 inches, and the corresponding dimensions of the vapor pipes *H* of the first three effects are 18 inches, 20 inches, and 46 inches. If it were not for this enlargement of these parts of the apparatus, the vapors would have to travel at a very high velocity, the entrainment of juice would be

excessive, and the vapors could not escape with sufficient rapidity.

9. Aside from the loss of sugar involved, entrainment is very objectionable, since the water obtained from the condensation of the vapors is used in feeding the steam boilers. When the feedwater contains an appreciable quantity of sugar, foaming is produced, and this may endanger the safety of the boiler.

The upper part of the steam space of each calandria is connected by means of a small pipe, which is provided with suitable regulating valves, either with the vapor pipe of the last effect or with its own vapor pipe. The purpose of these pipes is to remove the ammoniacal gas that results from the decomposition of certain nitrogenous constituents of the juice. The gas is lighter than the steam; hence it rises to the upper part of the calandria. The quantity of ammoniacal gas produced in evaporating cane juice is very small, but the contrary is the case in treating beet juices. The quantity of the gas in beet-sugar manufacture is so large that where proper provisions for its removal are not made, the tubes of the evaporator may be destroyed.

10. The remaining details of the construction of the quadruple effect will be explained in the following description of the manipulation of the apparatus:

Referring to Fig. 2, the travel of the juice and vapors follows the order of the Roman numerals. To begin work with the apparatus empty, the vacuum pump is started and juice is admitted into vessel *I* through pipe *G*, into *II* through pipe *c*, and so on. The pipes *G* and *c* are provided with valves so as to regulate the flow of the juice. Having filled each pan with juice to a point a little below the tops of tubes, steam is turned into the calandria of the first pan through the valve *F*. The steam used for this purpose is that exhausted by the various engines of the factory. Sufficient pressure is secured by the use of a back-pressure valve, and deficiencies in the supply of exhaust steam are made up with live steam. The juice in the first pan soon begins to boil,

and the vapor generated passes through the vapor pipe and into the calandria of the second pan, where it is condensed in boiling the liquor in this vessel; in a like manner, the juice is boiled in the third and fourth pans. The vacuum in the last pan is produced by the condensation of the vapors from that vessel and the assistance of the vacuum pump; that in the second and third pans, by the condensation of their vapors in the calandrias of the third and fourth pans, respectively; and in the first, or pressure, pan, as it is often termed, the pressure or vacuum maintained is regulated by the amount of steam admitted at the valve *F*.

The condensation water from the calandria of vessel *I* flows through the pipes *a* and a steam trap to the feedwater tank of the boilers; that from the remaining pans is removed by means of pumps.

11. The evaporation having been started as described, the level of the boiling liquor in each vessel should be maintained high enough to just cover the tubes of the calandria. This is done by adjusting the flow of juice through the pipes *G* and *c* by means of the valves at each pan. The sirup is pumped from the last pan through the pipe *d* into the storage tanks. The flow of sirup to the pump is regulated by a valve in pipe *d*. This valve is opened or closed a little at a time until sirup of the desired density is obtained. It should be noted that a current of juice flows through the apparatus from pan to pan, fresh juice entering at the pipe *G* and finished sirup being pumped out of the last vessel at *d*. The process is therefore a continuous one.

The steam pressure in the calandria of vessel *I* is kept as low as is consistent with obtaining sufficient duty from the apparatus and maintaining a regular flow of liquor from pan to pan. Under ordinary working conditions, the gauges connected with the vapor spaces of the vessels will show the following indications: *I*, 2 to 3 inches (mercury) pressure; *II*, 5 to 7 inches vacuum; *III*, 15 to 16 inches vacuum; *IV*, 27+ inches vacuum. The vacuum in the fourth pan should be as high as the condenser and pump will produce. The

pressure in the first pan and the vacuums in the second and third are regulated by the admission of steam to the calandria of the first pan.

The vacuum conditions are somewhat different in operating a triple effect. In such a case, the gauges show, respectively, 3 to 5 inches vacuum, 15 to 17 inches vacuum, and 27+ inches vacuum.

12. Chapman's Circulators.—In order to promote the circulation of the liquor through the tubes and to increase the capacity of the evaporator, **Chapman's circulators** are frequently used. In installing the circulators, a diaphragm is placed at the bottom of the down-take *E*, Fig. 2, of each vessel, and a tube, forming an inverted siphon, connects the space above the diaphragm of one pan with that below the diaphragm of the next vessel. The pipes *G* and *c* are not used. The space above the diaphragm of the fourth pan is connected directly with the sirup pump. The clarified juice is delivered to the first pan, below the diaphragm, by means of a pipe provided with a regulating valve. Since there is more or less steam formed as the juice passes from pan to pan, due to the difference of vacuum, a separating device is used, which insures the delivery of the juice free from vapor, where required. The height from the top of the calandria to the lowest part of the siphon must be such that the latter will always be filled with liquor, and thus prevent the vapor from checking the flow of juice.

13. The operation of the apparatus may be described as follows: Assuming that the pans have been charged with juice, the liquor boils up through the tubes *D* of vessel *I*, overflows into *E*, and is carried by the siphon to the lower compartment of *II*; it then boils up through the tubes of that pan, overflows as before, and passes through the siphon to pan *III*; and so on. The liquor that overflows into the down-take of vessel *IV* is pumped to the sirup storage tanks. If the density of this sirup is too high, the attendant increases the flow of juice into the first pan, and vice versa. When using the circulators, it is simply necessary to regu-

late the steam pressure in the calandria of the first pan and the quantity of liquor in that pan. If attention is paid to these details, the siphons will maintain the proper level of liquor in the other pans. The rapid circulation of the juice through the tubes of the calandria promotes the evaporation and reduces the fouling of the heating surfaces.

14. Formation of Scale and Its Removal.—All metal surfaces used in heating and evaporating cane juices rapidly foul, or scale. The material that deposits on the heating surfaces and bakes to a scale is a very poor conductor of heat and greatly reduces the efficiency of the apparatus.

TABLE I
COMPOSITION OF SCALE

Ingredients	1st Pan Per Cent.	2d Pan Per Cent.	3d Pan Per Cent.	4th Pan Per Cent.
Phosphate of lime	57.85	56.98	15.02	7.49
Sulphate of lime	2.02	1.92	.54	1.65
Carbonate of lime	3.25	4.68	19.55	9.93
Silicate of lime	7.86	13.31	.71	7.02
Oxalate of lime			11.32	11.27
Iron oxide	2.03	1.53	2.31	2.58
Silica	7.79	7.43	39.26	54.34
Combustible matter . . .	20.37	13.41	11.04	5.08

The tendency of the juice to form scale varies with the locality in which the cane is grown. In certain cane districts, a multiple effect can be operated only a week before it is necessary to clean its tubes, and in other localities, under the same manufacturing conditions, the heating surfaces scale slowly and the apparatus may be used 2 weeks or more without cleaning. In Table I is given the composition of the scale that formed in a quadruple effect in Java.

15. Comparatively little scale forms in the tubes of the first pan of the multiple effect, but this is not always the

case. The quantity increases from pan to pan as the liquor increases in density, and is always thickest in the last, or sirup, pan.

As will be noted by an inspection of Table I, the composition of the scale differs greatly in the different pans. The scale from the fourth pan, or effect, as the vessels are often styled, would evidently be more readily attacked by caustic soda than by muriatic acid, on account of its large silica and silicate content; this is also true of the scale from the third pan. The first and second effects contain a scale rich in substances that are soluble in muriatic acid, but the scale also contains a large proportion of organic matter. It is apparent from these analyses that boiling caustic-soda solution, followed by muriatic acid, should be very effective in the removal of the scale, and it is with these reagents that the evaporators are usually cleaned.

16. In practice, a strong caustic-soda solution, containing from $1\frac{1}{2}$ to 2 pounds of soda per cubic foot of water, is boiled in the pans for 2 or 3 hours. The soda solution is then run into a storage tank, for future use, and the heating surfaces are rinsed with water. The action of the soda softens the scales, but dissolves very little of this material. This soda treatment alone is often sufficient, but, usually, it is also necessary to boil dilute muriatic acid in the pans. Both the soda and the acid should be boiled with the pans open, that is, at atmospheric pressure. The spent acid is run into the sewer. The soda solution may be used many times by decanting it from the mud, which settles to the bottom of the tank, and occasionally adding fresh caustic soda to it.

The heating surfaces may also be freed from scale by means of tube scrapers. The scale should be kept wet with water during the scraping. This method of cleaning, however, requires so much time that it is usually only available when the manufacture is suspended for a period of several hours.

17. Purification of the Sirup.—The sirup as it comes from the vacuum evaporators contains much matter in sus-

pension that has been separated during the concentration and that was soluble in the thin juice. For the storage of the sirup and the removal of this precipitate, the liquor is run into settling tanks, and, after a suitable period of rest, it is decanted into the vacuum-pan charging tanks. The precipitate is disposed of as previously described.

Many sugar makers heat the sirup until it boils, then skim off the impurities that rise to the surface—that is, clarify it, —and finally run the liquor into settling tanks. As has been noted in *Manufacture of Sugar*, Part 1, sucrose is readily inverted by heat alone, but more rapidly in the presence of air. It is thus evident that great care is necessary in the clarification of sirup, since its boiling point in the open air is above 102° C. To avoid loss of sugar when clarification is practised, the heat should be discontinued when the liquid begins to boil. It is doubtful whether this process is advantageous, except in the manufacture of white sugar.

CRYSTALLIZATION OF SUGAR

18. The sirup obtained by the evaporation of the juice usually has a density* of 54.3° Brix at $17\frac{1}{2}$ C.—corresponding to 30° Baumé and 1.257 specific gravity—and contains about 46 per cent. of water. If only the matter of the further concentration of the sirup were involved, it could be readily and economically accomplished in the multiple effect; but the crystallization of the sugar must also be considered. In all modern factories, the sugar is crystallized, or grained, in a single-effect, vacuum evaporator, termed a *vacuum pan*, and this crystallization is effected while the liquor is boiling. This process requires that the sirup used shall be of moderate density, as will be shown later. The vacuum-pan process of crystallizing sugar has resulted in great economy in the manufacture and in the direct production of high-grade sugars.

* In sugar factories, the word *density* is used synonymously with *specific gravity*, and is expressed either in degrees Brix or in degrees Baumé

The apparatus used and the methods of procedure followed in "boiling" vacuum-pan sugars will now be described.

19. Vacuum Pan.—A vacuum pan, shown in Fig. 3, consists of a cylindrical cast-iron or copper vessel with a conical bottom and dome-shaped top. Copper, however, is not often used in this construction. A vapor pipe *A* leads from the dome to a save-all *B*, and from this point to a vacuum pump. A large valve, called the *strike*, or *foot-valve*, is located at the bottom of the pan at *C*. This valve is used for discharging the *massecuite*; that is, the mass of sugar crystals and molasses. A series of copper coils—usually four or more in number—fitted with suitable valves *D* forms the heating surface. A large opening, or well, similar to the down-take of the multiple effect, is provided at the centers of the coils. This well serves to promote the circulation of the *massecuite* and to give access to the pan for repairs. The coils are drained of condensation water through tail-pipes and steam traps. A sirup charge pipe *E* enters the pan either near the strike valve or at a higher point, and in the latter case, passes downwards to near the bottom of the vessel. This pipe has branches that connect with the molasses tanks. Eye-glasses *F* for watching the progress of the work, a proof stick *G* for withdrawing samples of the material for examination, a vacuum gauge *H*, a thermometer *I*, a break-vacuum valve *K*, and a steaming-out pipe *L* complete the usual equipment of the pan.

20. Many factories have a central condenser with which all the pans and multiple effects are connected. With this type of installation, a head-valve is placed in the vapor pipe, to be closed when the pan is not in use. A small pipe connects the pan with the vacuum line, for the purpose of establishing the same pressure on both sides of the head-valve when a "strike" of sugar is to be boiled. Pans are also occasionally arranged for the discharge of the *massecuite* by air pressure and its distribution among the crystallizers through large pipes. This arrangement also requires a head-valve in the vapor pipe.

21. There are two types of pumps employed to produce the vacuum, namely, that of the wet system and that of the dry system. In the wet system, the water of condensation from the vapors and the condensing water are passed through the pump. In the dry system, the condensation and condensing waters are led off through a Torricellian tube, or "leg pipe," the lower end of which dips below the surface of water into a small tank, called the *hot-well*. The water in this well seals the pipe and at the same time permits the escape of water from the condenser. A small pipe connects the condenser with the vacuum pump, which removes the air from the pan when beginning an operation, or strike, and also the air that enters the apparatus by leakage or that which is entrained by the sirup.

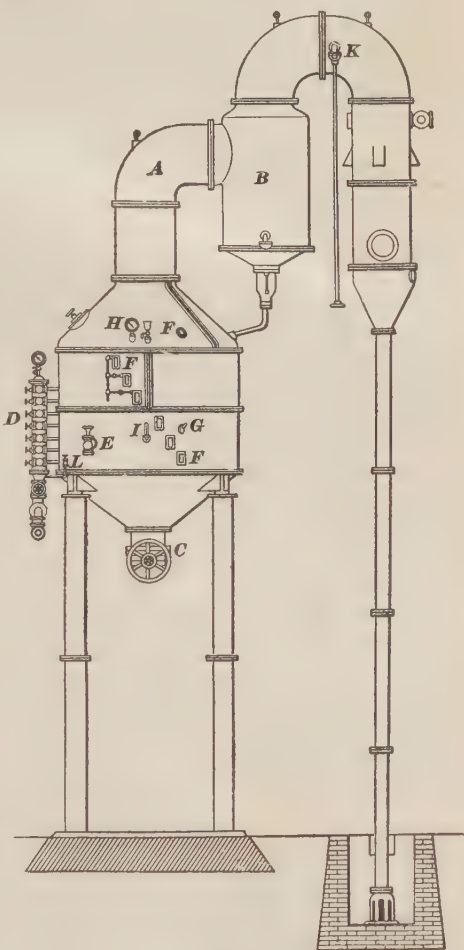


FIG. 3

The degree of exhaustion of the pan, or, to use the usual sugar-house expression, the *vacuum*, is increased or decreased by increasing or decreasing the quantity of water injected

into the condenser. This regulation of the vacuum is of importance, since the temperature of the massecuite must be varied at different stages of the operation of boiling the sugar. When a common condenser is used for several pans, the vacuum is regulated by the head-valve, which must be slightly closed when it is desired to reduce the vacuum.

The heating surfaces of the vacuum pans foul, or scale, but not so rapidly as those of the multiple effect. These surfaces may be readily cleaned by boiling a strong caustic-soda solution in the pan.

22. Vacuum-Pan Process of Boiling Sugar.

Having described the apparatus used in the boiling of sugar—the vacuum pan—the methods will next be considered. While it is important that the process of sugar boiling and the principles on which it is based be thoroughly understood, nevertheless, to become proficient in this work, actual practice at the vacuum pan is essential. Practice is necessary in order to learn to judge the relative densities of the liquor under different conditions of purity, vacuum, and temperature, from the appearance of the material as it is boiling in the pan, and from the examination of test samples drawn with the proof stick. Practice is also necessary in order to decide on the necessary manipulations from the quantity and appearance of the crystals in the test samples.

Factory terms will be used in the following descriptions of methods, but will first be defined. The essential parts of the vacuum pan have already been described.

TERMS USED IN SUGAR BOILING

23. Sirup.—The clarified juice of the cane evaporated to a density of about 54° Brix. Sirup* of other densities may be used, but that indicated is customary in good practice.

Grain.—The sugar crystals in the massecuite. This word is also used as a verb, to indicate the process of forming the crystals.

*The refiners apply this word to the final molasses.

Proof.—That stage of the process when the liquor has been concentrated to a state of supersaturation with sugar. In boiling strikes of molasses, the word is modified to “string-proof,” “hook-proof,” etc., according to the density of the material.

Boiling to Grain.—The boiling of a strike in which the sugar is crystallized in the vacuum pan.

Boiling Blank, or to String Proof.—The boiling of a strike in which the crystallization of the sugar is left to a subsequent operation.

Massecuite.—The thick mixture of sugar crystals and the mother liquid; also, where strikes are boiled blank, or to string-proof, the heavy material, ready for the subsequent crystallization of the sugar, is called *massecuite*. The word is also, for convenience, applied to the material in the pan at all stages of the process. The massecuites are numbered serially, according to their origin, beginning with that from sirup, which is called *first massecuite*.

Mixed Massecuite.—A massecuite formed partly of sirup and partly of molasses. The molasses is said to be “boiled in.”

Molasses.—The liquid residue left after removing the sugar from the massecuite. The various grades of molasses are numbered to correspond with the massecuites from which they are obtained.

Strike of Sugar, or Massecuite.—The completed massecuite from a boiling is termed a *strike*, and the operation is called “boiling a strike of sugar, or massecuite.” The word strike is also applied, in the same way, to molasses.

Cut.—The part of a strike that is left in a pan as a nucleus on which to boil another strike. This word is also used as a verb; in this sense, the English very often use the word *double*. Many sugar makers use the word cut to indicate the portion of the strike that is removed from the pan.

Cut-Over Pipe.—A pipe used to transfer a portion of an unfinished strike from one pan to another, each portion serving as a nucleus on which to boil a cut strike.

Final Molasses.—The liquid residue from which more sugar cannot usually be profitably extracted.

Circulating, or Condensing, Water.—The water injected into the condenser.

METHODS OF SUGAR BOILING

24. Grained Strikes.—Having closed the foot- and break-vacuum valves of the pan, the vacuum pump is started and the circulating water is turned into the condenser. When the gauge indicates a vacuum in the pan of from 15 to 20 inches, the sugar boiler opens the charge valve and draws sirup into the vessel until the copper coils are covered, or until, from previous experience, he judges that the pan contains sufficient liquid. He now carefully and slowly opens the steam valves, one at a time, beginning with the lowest, or No. 1, as it is called, allowing time for the water of condensation to escape from the coil. Care is necessary, so as to avoid injuring a coil. The sirup soon boils and the water evaporates rapidly from it. When the surface of the liquor falls to near the top coil, the steam valve on that coil must be closed, and so on from coil to coil, never leaving one uncovered while its steam valve is open.

Soon, from the appearance of the sirup and from the flow of the drops projected on the eye glasses, the sugar boiler notes that the liquor is becoming very dense. By gradually closing the circulating-water valve, he raises the temperature of the boiling liquid to about 65° C., or to such other temperature as the grade of sugar he is boiling requires, and by manipulating the water valve maintains this condition. From time to time, a test sample is drawn with the proof stick and is examined on a piece of plain glass by means of transmitted light. In a short time, the boiling liquid becomes saturated and then supersaturated with sugar, at the pan temperature, and microscopic crystals of sugar begin to form.

It requires experience on the part of the pan boiler to detect promptly these minute crystals, which an inexperienced eye would scarcely note. The sugar boiler now carefully

follows the formation of the crystals, and the moment he judges that these are numerically sufficient, he opens the charge valve and draws a small quantity of sirup into the pan; he also slightly closes the circulating-water valve so as to prevent a fall in the temperature of the boiling liquor. A large charge of sirup would cool the boiling mass and thus cause additional crystals to form, and the same thing would occur if the vacuum should rise and thus cool the liquor. The reason for this is that when a solution is saturated with sugar at a certain temperature, it is supersaturated at a lower temperature and the crystals separate.

25. As the charge of sirup evaporates, the panman carefully watches the liquid until it is again saturated, but he avoids supersaturating it by injecting another charge of sirup into the pan. By this procedure, he forces a part of the sugar in the sirup to deposit on the crystals already present, instead of forming new ones, or *false grain*, as this condition is termed. The sugar boiler frequently examines test samples on the piece of glass, and searches for false grain, which he must immediately get rid of if produced. This is accomplished by increasing the temperature of the pan and charging copiously with sirup. The moment the disappearance of the false grain is noted, the panman begins to cool the massecuite gradually, and when the temperature falls to about 65° C., he continues the boiling as before. False grain is objectionable, because it impedes and often obstructs the centrifugating of the sugar. A loss of sugar also occurs by the fine crystals passing through the centrifugal sieves.

The alternate charging and evaporating are continued until the pan is filled with massecuite. The charges of sirup are gradually increased in volume until the pan is about two-thirds full, when they are decreased. During the latter stage of the operation, the massecuite is gradually increased in density from charge to charge, and finally it is evaporated to a water content of about 7 per cent. During this process, the crystals increase from a minute size to that of commercial sugar.

The steam is now shut off from the coils, the vacuum pump is stopped, and the break-vacuum valve is opened. When the vacuum falls to zero, as indicated by the gauge, the massecuite is discharged from the pan through the foot-valve. The further treatment of the massecuite is described in the paragraphs devoted to crystallization in motion and to curing the sugar.

After boiling and discharging a strike, the pan must be thoroughly cleaned by steaming, to recover the last adhering portions of massecuite. This steaming is necessary not only to recover the sugar, but also to prevent it from caramelizing on the coils.

26. At the beginning of the strike, it is important that the pan boiler form a sufficient number of crystals and that he at all times avoid excessively large charges of sirup, otherwise false grain may be formed.

The grade of sugar required and other considerations determine the point in the pan at which the strike is grained. When the crystals are formed between the first and second coils, the strike is said to be *grained low*, and when formed near the middle of the pan, to be *grained high*. Low grain-ing produces comparatively few crystals of large size, and requires long boiling. High graining, on the contrary, produces a fine-grained sugar, with short boiling. When a strike is to be cut over from one pan to another, or an ordinary cut is to be made, except in the production of large crystals, it is usually grained high.

The conduct of the work is necessarily modified somewhat by the use of low-purity sirups or by the boiling in of molasses. With this class of materials, the strike is usually boiled at a high temperature and is "kept close," that is, very dry. When boiling very rich sirups, it is necessary that the strike be "kept very free," or of moderate density.

27. The temperature conditions vary in boiling different grades of sugar. A soft, low-test sugar is produced by boiling the strike at as low a temperature as possible, and clean, hard crystals are obtained by hot boiling. Large crystals

are more readily obtained with thin sirup and the consequent slow evaporation. The commercial granulated sugar is boiled at a high temperature when produced in the factory, and usually also when made in the refineries.

The large-grained sugar of a light-yellow color, known as *Demerara crystals*, is colored in the vacuum pan in the boiling process by means of dilute sulphuric acid. This sugar is made in Demerara, for the British market. The large crystals are usually obtained by "cutting," or "doubling." This is a wasteful process, however, because the acid inverts much of the sucrose of the sirup. Muriate of tin (stannous chloride) is used both in the vacuum pan and in the sugar wash in the centrifugals to obtain a bright or a yellow product.

28. The methods of graining and charging that have been described are not the only ones used. Many sugar boilers, in graining, concentrate the sirup to a certain density and then increase the vacuum, thus cooling the dense sirup until it is supersaturated with sugar and causing the crystals to separate. A small charge of sirup is drawn into the pan at the time of lowering the temperature. This operation is repeated until sufficient crystals are obtained. With many, but not with all installations, the charging may be continuous instead of intermittent. In this method, the charge valve is opened immediately after graining, and from then until the end of the strike, its opening is so regulated from time to time as to maintain a saturated sugar solution.

29. Cut Strikes.—When a **cut strike** is to be boiled, a part of the massecuite from a previous strike is left in the pan, and its crystals take the place of the grain that would otherwise have to be formed. Sufficient massecuite must form the cut so as to supply enough crystal surface for the deposition of the sugar in the sirup, otherwise false grain will result; or, if this is avoided, the molasses surrounding the crystals will not be satisfactorily exhausted of sucrose. This method produces a very large-grained sugar.

30. Blank Strikes, or Crystallization at Rest.—In sugar boiling, the terms *string strikes*, *smooth strikes*, and *jellied strikes* have practically the same meaning as **blank strikes**. Until a few years ago, the only grained sugar boiled was that of the first crystallization, leaving the molasses to be boiled blank. In boiling blank strikes, the molasses is first heated so as to melt the fine crystals it usually contains; it is then drawn into the vacuum pan and is concentrated under as high a vacuum as can be obtained with the apparatus. The density to which the massecuite is concentrated depends largely on the richness of the molasses. The evaporation is usually stopped when a test sample held between the thumb and finger produces a strong thread when these digits are separated. The test is often made by pouring the test sample from the proof stick and noting the appearance of the *string* when it breaks. The temperature of the massecuite at this stage should be as low as possible, and, with an efficient installation and good molasses, is usually about 68° C.

31. Considerable skill is required in boiling blank strikes, since the panman must properly judge the quality of the material largely from its appearance, and must apply the crude test described under varying conditions of purity and temperature. The strikes boiled to a "string-proof" are usually run into small, rectangular sugar wagons or into large tanks to accomplish the crystallization. The wagons of massecuite are placed in a hot room and are allowed to remain there a week or longer at a temperature of about 38° C., this time depending on the purity of the molasses. This method may be termed *crystallization at rest* to distinguish it from the more modern method of *crystallization in motion*, which will be described further on.

Massecuites boiled blank and grain strikes of low purity often foam in the wagons, or tanks. This condition is probably due to a decomposition of the compounds formed during the processes of manufacture. Sugar boilers usually attribute the foaming to the boiling of the massecuite at high

temperatures. When foaming occurs, water should be poured on the surface of the massecuite and the foam then beaten down with a paddle.

32. The products made from blank strikes are often termed *molasses sugars*. These sugars have a low polariscopic test, or polarization, which usually ranges from 80° to 90°. Two grades of molasses sugar are usually made, namely, *seconds* and *thirds*. The third sugar is boiled at the close of one grinding season and left until the beginning of the next, or for a period of 2 or 3 months, before centrifugating.

33. Crystallization in Motion.—The process of **crystallization in motion** was devised in the beet-sugar industry by Wulf, in 1884, and was made a practical process by Stammer and Bock, and it is now extending rapidly in cane-sugar manufacture. While the process possibly does not secure a larger yield of sucrose in the sugars than does the ordinary process, namely, crystallization at rest, it produces as much in a single grade of high-test sugar.

Crystallizers will be described fully in connection with beet-sugar manufacture. For the present purpose, a brief description of the apparatus will suffice. The usual type of crystallizer is a horizontal iron cylinder that has both ends closed and is provided with a charging door, a discharging valve, and a spiral mixer. The cylinder is often fitted with a water-jacket for regulating the temperature of the massecuite.

34. There are several methods of using the crystallizers, the simplest of which is to run a strike, boiled blank, into the apparatus and to mix it, by means of the spiral, for several days, or until there is no further crystallization of sugar. This method produces low-grade sugar and is seldom used.

35. In the usual method, a strike of first sugar is boiled to grain, sufficient diluted molasses being used to so lower its coefficient of purity that it will yield a molasses, on

purging, having a purity of about 55°. This massecuite is usually either centrifugated immediately on leaving the vacuum pan or kept in motion in the crystallizer for several hours. A mixed strike of massecuite is next boiled, using grain formed from sirup or a small quantity of first massecuite as a nucleus, or footing, for the strike. The 55° purity molasses from the previous and other strikes is diluted to 30° Baumé and is boiled in on this footing to form a massecuite of about 73° purity. This massecuite is run into a warm crystallizer and is slowly stirred for from 3 to 4 days, or until the molasses from a sample of it, purged in a laboratory centrifugal, has an apparent coefficient of purity of about 47° or somewhat lower. The massecuite is then centrifugated, using a little wash on the sugar if necessary. A third strike is boiled, using sirup or first massecuite, as before, for a nucleus on which to build the crystals, and the 47° purity molasses is diluted and boiled in so as to form a massecuite of about 60° purity. This massecuite is also run into a warm crystallizer, and is kept in motion as long as is practicable, or until the analysis of test samples shows that the molasses is no longer decreasing in purity. The sugar is then purged.

The temperature of the massecuite in all cases is permitted to fall gradually, often to nearly that of the factory. As the massecuite cools, the sugar is forced out of solution, and owing to the circulation of the crystals through the mass, it deposits itself on these, instead of forming new ones. It is sometimes necessary to dilute the massecuite in the crystallizer a day or more before purging, in order to facilitate the separation of the sugar. At times, this dilution is also advisable when the massecuite has been evaporated until very dry, and the molasses should be added for this purpose after a fall in temperature of a few degrees, otherwise false grain may form. The object of warming the crystallizer before use is to prevent the formation of crystals when the massecuite comes in contact with the metal.

The molasses used for boiling in or for diluting should be heated to free it from crystals of sugar, and for the former

purpose should be used at a temperature about the same as that of the massecuite in the pan.

36. Another method of using a crystallizer is to boil a strike of grained sugar in the usual manner, and having reduced the massecuite to a very low water content, to draw into the pan sufficient hot molasses of about 48° Baumé, little by little, to reduce the massecuite to the desired coefficient of purity. This mixture is now run into a crystallizer and is stirred as usual.

Numerous slight modifications of these methods are used, but those described will answer the present purpose (see also *Manufacture of Sugar*, Part 3). These descriptions refer to raw-sugar manufacture. With Louisiana cane, the work may be conducted with somewhat lower purities of massecuites and molasses than those stated, which apply in Cuba. The tropical-cane molasses of low purity is exceedingly viscous and has a glucose coefficient of about 50, whereas this coefficient often passes 100 in Louisiana molasses. Possibly this condition accounts for the low molasses purities readily obtained in Louisiana.

The process of crystallization in motion not only enables the manufacturer to produce high-test sugars, but also to do so promptly and with little labor. Crystallization at rest necessitates a very large installation of tanks or cars. With this process, rich massecuites require from 7 to 10 days, and the very low grades from 1 to 3 months, for the crystallization of the sugar, as compared with from 3 to 5 days for all grades where crystallization in motion is used.

CURING THE SUGAR

37. Separating Crystallized Sugar From Molasses. The crystals of sugar must be separated from the molasses that surrounds them either by simple drainage or by an application of centrifugal force. The removal of the molasses is termed *purging*, and where centrifugal machines are used it is often called *centrifugating*. In the old open-kettle

factories, the room devoted to the drainage of the sugar was called the *purger*.

All factories formerly purged the sugar by simple drainage. In this method, the massecuite is packed in hogsheads having perforated bottoms, over which cane leaves or other loose material is placed. Sugar canes are also inserted in the massecuite so as to lead the molasses to the bottoms of the hogsheads. The molasses drains into cisterns over which the hogsheads are placed. The sugar obtained by this method in open-kettle factories is called **muscovado**. The molasses is of very good flavor, and in the United States is called **Louisiana**, or **open-kettle**, molasses. Only small quantities of muscovado sugar and open-kettle molasses are now produced.

In Java, simple drainage is used to remove the molasses from very low-grade sugar having fine crystals. The massecuite is packed in bags, or mats, through which the molasses drains. The resulting sugar contains much molasses and is called *sack sugar*.

38. Centrifugals.—Sugars are separated from the molasses in the modern factories by means of machines termed **centrifugals**. The centrifugal is essentially a perforated metal basket, lined with metallic gauze, and is so arranged that it may be charged with massecuite and then be rapidly revolved. The massecuite distributes itself on the lining, owing to the centrifugal force, and its molasses passes through the perforations, leaving the crystals in the machine.

39. Types of Centrifugals.—Centrifugals are of two types, namely, the *Weston*, or *suspended*, machine, and those centrifugals in which the lower end of the vertical shaft, or spindle, is supported by a bearing. The Hepworth machine is an example of the latter type. The Weston and Hepworth types of centrifugals are most generally used in the cane-sugar industry. The baskets are usually made 30, 36, and 40 inches in diameter.

40. Operation of Centrifugals.—The operation of a centrifugal will be described so that the production of the various grades of sugars may be more clearly understood. The basket of the machine is charged with massecuite either while it is at rest or while it is in motion, according to the condition of this material or the preference of the workman. The quantity of massecuite forming a charge depends on the depth of the ring at the top of the basket and also on the quality of the material. With a free-purging massecuite, the charge should be large enough to form a wall, when the machine is in motion, the depth of the ring in thickness. With gummy massecuites, this thickness must be greatly reduced. Having charged the machine, its speed is quickly increased to that for which it is designed. The speed varies with the diameter of the basket. A 30-inch basket is usually driven at the rate of 1,200 revolutions per minute, while the larger sizes are driven at a lower number, but at the same periphery speed. As the speed increases, the molasses leaves the crystals, passes through the perforations of the sieve, or lining, strikes the outer casing of the machine, and then drips into a gutter.

41. If unwashed raw sugar is to be made, the workman stops the machine and discharges the sugar from the basket through an opening at the bottom just as soon as he notes that no more molasses is being thrown off. In using certain European makes of centrifugals, the sugar is lifted from the basket by means of a paddle. This sugar is almost free from molasses and is ready to be packed for shipment.

If the sugar that is dried as just described does not test high enough with the polariscope, its polarization may be increased by washing it while it is still revolving in the machine. When the sugar is to be washed, the workman watches for the disappearance of the molasses from the face of the wall, and then sprays a small quantity of water or other wash on it from a special form of syringe or some other device. The water softens the molasses and washes it from the surfaces of the crystals.

42. In making white sugars, they are washed as just described, except that large quantities of water and a blue wash are used. The blue wash, usually prepared with ultramarine, is for the purpose of "killing" the slight yellowish tinge of color that usually persists in white sugars made directly from the cane.

43. Yellow sugars are brightened, or their color is heightened if need be, by a wash containing muriate of tin (stannous chloride). Even if the color of the sugar at the time of purging is satisfactory without the tin wash, it would not long remain so. The crystals would soon lose their brightness. The quantity of the tin salt that adheres to the sugar is an extremely faint trace, but it is sufficient to insure the keeping qualities of the product.

44. **Granulation of Sugar.**—White sugar is often submitted to a drying or granulating process after it leaves the centrifugal. This drying apparatus is termed a *granulator*, and the product is the **granulated sugar** of the markets. The granulator not only dries the crystals, but, as its name implies, separates them from one another.

This apparatus is a long iron cylinder about 5 feet or more in diameter, and is so arranged that it may be slowly revolved in a slightly inclined position. Angle irons, forming shallow shelves, are attached to the inner surface of the cylinder at short distances from one another and extend lengthwise of it. An exhaust fan is located at the sugar inlet end of the apparatus, and serves to draw a current of air through a steam heater and thence through the granulator. The ends of the apparatus are boxed in so as to direct the air to the fan.

45. When the granulator is in operation, an elevator delivers an almost continuous stream of moist sugar through a chute into the higher end of the revolving cylinder, and the shelves pick up the sugar and carry it upwards until it slips from them and falls in a shower through the current of hot air. This operation is repeated until the sugar leaves the cylinder at its lower end, owing to the inclined position

of the apparatus. In its travel through the granulator, the hot air abstracts all the moisture from the sugar, and the motion of the latter separates the crystals from one another. On leaving the granulator the sugar is sifted, to remove lumps, and, in the refineries, to separate it into the *fine*, *standard*, and *coarse granulated sugars* of the markets.

There are several types of granulators, but as the principle on which they are based is the same for all, one of the simplest has been described.

46. The moist sugar may be pressed into molds, so as to form large loaves when subsequently dried. It may also be moistened with white sirup and then pressed into cubes by means of a special machine. The cubes are dried in an oven before packing.

The residual molasses is generally utilized in the manufacture of alcohol or in feeding the draft animals on the plantation.

MANUFACTURE OF BEET SUGAR

(PART 1)

PRODUCTION AND STORAGE OF BEETS

1. The manufacture of beet sugar has been practiced in Europe for more than a hundred years; in fact, nearly all the sugar consumed there is obtained from this source. The beet-sugar industry in the United States, however, is of very recent date. The sugar produced prior to 1889 was derived almost exclusively from the tropical cane in Louisiana. The refining branch of the industry depended on foreign sources for nearly its entire supply of raw sugars, which was obtained from both cane- and beet-sugar producing countries.

2. Climatic Conditions.—The most favorable climatic conditions for the sugar beet are found where the mean summer temperature is 70° F. Dr. William McMurtrie traced a mean isotherm of 70° F., based on observations made during 10 years for the months of June, July, and August, and considered the beet area to be in the vicinity of this line, where the minimum rainfall for the three summer months is 2 inches per month. At the instance of Dr. H. W. Wiley, this isotherm was retraced, and from this and other data he concluded that the American sugar-beet area is within a belt extending 100 miles each side of the 70° F. isotherm. Doubtless, there are many localities near this belt where the conditions are favorable to the beet.

Growing conditions for sugar beets are found in a wide belt of the moderate climatic zone. In Europe, sugar beets are grown as far south as the northern parts of Spain, and Italy, and as far north as Sweden, and Norway, that is, in the belt

between about the forty-second degree of latitude and about the sixtieth degree of latitude. The Gulf Stream is probably responsible for the moderate climate of the latter countries. In North America, sugar beets are grown from as far south as Southern California to as far north as Montana, Washington, and Ontario, Canada—that is in the belt between about the thirty-third degree of latitude and the forty-eighth degree of latitude.

The sugar beet requires an abundance of sunshine for its proper development as a source of sugar; hence, northern latitudes usually produce the richest beets. The plant grows luxuriantly in the South, but the roots rarely contain paying quantities of sugar. Experience has demonstrated that beets of satisfactory richness are not produced very far south of the belt defined by Wiley.

The question of rainfall is also of great importance. Summer rains are very desirable, but heavy autumn rains, especially following a dry summer, are extremely unfavorable to the sugar content of the beet. Late rains cause a second growth of the plant at the expense of the sugar. Cold winters, without alternate freezing and thawing, are desirable for the proper keeping of the roots in storage preparatory to the manufacture.

To recapitulate, Doctor McMurtrie stated the favorable meteorological conditions to be in general, "Comparatively dry and warm spring months during the time for preparation of the soil, planting, and cultivating the crop; moderate temperature, abundant and frequent rains during the summer months * * * ; cool, dry fall, the time for ripening, harvesting, and storing the crop."

3. Varieties of Beets.—The several varieties of sugar beets now known have been derived by selecting roots that possessed valuable sugar-producing qualities. All the varieties are similar to those of the common garden beet, or *Beta vulgaris*.

Among the first, at least in France, to make scientific investigations and experiments for the selection of rich

varieties of beets and for fixing these qualities was Louis de Vilmorin, who was ably followed in this work by his son, Henri de Vilmorin. The roots for producing seed were selected for their shape, size, and color, the character of their foliage, their keeping qualities, and their high sugar content. Many investigators in various parts of Europe,



FIG. 1

and recently in the United States, have continued these and other investigations, until there are now many valuable varieties whose characteristics are fixed.

Among the varieties grown are Vilmorin's Improved White beet, French Very Rich, Klein Wanzlebener, White Imperial, and many others. Many of the very rich beets have been derived from Vilmorin's Improved and the Klein

Wanzlebener by systematic selection. Different varieties are chosen by the beet growers for planting under different soil and climatic conditions.

4. Typical Beet.—A typical beet, so far as shape and foliage is concerned, is shown in Fig. 1. This beet is large and tapering, with only a small neck, or crown, and with broad, spreading foliage. These conditions of shape facilitate harvesting. The long tap root penetrates the soil deeply in search of moisture; the spreading leaves shade the adjacent soil, thus conserving the moisture and preventing the growth of weeds; and the small neck, which must be removed in harvesting, results in little waste.



FIG. 2

Beets weighing more than 3 pounds are usually of inferior quality, but this depends more or less on the variety of seed, the suitability of the soil, and the conscientiousness with which the latter has been prepared and cultivated. As a general rule, however, the larger beets are poorer in quality, so far as the sugar content is concerned, than the smaller or medium-sized ones. The beets most eagerly sought by sugar manufacturers are the ones that weigh between 1 and 2 pounds. Where beets are too large or too poor in quality to be used in sugar manufacturing, they must not be considered entirely valueless, as they may be well and advantageously utilized as feed for livestock. Fig. 2 illustrates a cross-section of a beet, the dotted lines showing the concentric rings of growth.

5. Planting.—The beet seed should be planted as early in the spring as weather conditions will permit. The quantity of seed required per acre varies greatly. The seed germinates more quickly in some soils than in others and many things unite to act against the germination of every seed, so that it has been found best to plant enough to insure a good stand and also to supply the force necessary to lift the crust of the

soil and to allow the tender sprouts to reach light and air. It is generally conceded that each acre of land requires from 12 to 20 pounds of seed. The rows should not be less than 18, nor more than 20, inches apart, this space being sufficient for a horse to walk in during cultivation and permitting the leaves of the full-grown plant to shade the ground, thus avoiding rapid drying of the soil. The proper depth for planting depends very much on the locality. Where the atmosphere is humid and the ground moist, from $\frac{1}{2}$ to 1 inch is deep enough to lay the seed, while in very arid regions, $1\frac{1}{2}$ inches is necessary, owing to the rapid evaporation of the surface moisture. Where conditions permit, shallow planting is certainly advantageous.

6. Thinning.—When the seed has sprouted about 2 inches above the ground and the plants show four leaves, it will be found that they stand too close together. It then becomes necessary to thin them so that the remaining ones will be a uniform distance apart. This is done most economically by using a hoe that is 4 inches wide and has a sharp point. Spaces are struck out with the hoe, and small bunches of beets, from 6 to 8 inches apart, are left for two or three thinners, who deftly pull out all but the healthiest beet. Only one beet is left in a place, since otherwise the roots twist about one another and in the end none of the beets shows a healthy growth or is really worth harvesting. In poor soil, the beets should be thinned until about 10 inches apart; in fairly rich soil, 8 inches is preferable; and in very rich soil, the distance can be shortened to 6 inches. Large beets, as has been stated, are not the most desirable; hence, in thinning the plants, care should be exercised to avoid allowing too much space to each. As a rule, the plant will look weak and sickly after thinning, and generally about 48 hours is required before it recovers sufficient strength to hold up its head again.

7. Cultivation.—The sugar beet is a plant that requires plenty of nourishment, and it is to the interest of the beet raiser to keep the soil as free as possible from weeds. Cultivation should begin soon after the rows are well defined,

and should be repeated frequently. The old-fashioned ground cultivator is not suitable for this purpose, but there are numerous beet cultivators to be had. The Moline beet cultivator is considered to be one of the most efficient and is extensively used at present. Whatever cultivator is employed, the soil should be kept mellow and free from weeds. The crown of the beet, from the point where the foliage sprouts, should not be covered by the soil, nor should the beet be exposed too much. Laceration of the leaves should be avoided after they are large enough to protect the exposed part of the beet from the heat of the sun, and from this period until the time of harvesting arrives, it is desirable to keep the field free from weeds by hand. Experience has shown that one man is well able to keep a field of 20 acres clean.

8. Harvesting.—The exact time of maturity of the beets can only be determined by chemical analysis, but experience has demonstrated that when the period of maturity arrives, the dark-green color of the leaves disappears, the outer leaves wither and die, and the center leaves assume a reddish, golden-yellow color. Beets seeded very late in the season often fail to show these characteristic marks of maturity, yet the analysis shows a high percentage of sugar with a corresponding high coefficient of purity. When the chemist discovers that the beet is at its highest sugar content and coefficient of purity—that is, from 14 to 20 per cent. sugar, with a coefficient of from 80 to 85—the crop should be harvested as quickly as possible.

The factory arranges its contracts so that the delivery of beets can be made with regularity, and that all roots may be out of the ground before it freezes so hard as to prevent harvesting. The beets should be harvested before there is danger of a second growth.

When the beets are mature, they are loosened either by means of a strongly built plow or by a special harvester that lifts them. The plow or the harvester breaks the tap root and permits the beet to be readily removed from the soil by means of its leaves. After removing the beet from the ground,

it is *topped*; that is, the neck is removed at the lowest leaf scars with a strong knife. The neck, or crown, of the beet contains comparatively little sucrose and a large proportion of mineral matter that is objectionable in the manufacture of sugar. The tops are either left to rot in the fields, or pastured, thus returning considerable valuable fertilizing material to the soil. After topping, the beets are thrown into heaps, and if not immediately removed to the factory, they are covered with leaves to protect them from the sun.

9. Wide-tired wagons are used to convey the beets from the fields to the cars or storage sheds. There are several dumping devices in use to lessen the labor of unloading the wagons. Among the simplest of these is a tilting platform on which the wagon is clamped and then inclined to discharge the beets. This method is used where the beets reach the sheds comparatively free from adhering soil.

In certain localities, notably in California, the beets are loaded on a strong net spread over the wagon. When ready to discharge the load, one side of the net is fastened to the car, or bin, and ropes attached to the free side are passed over the car, or bin; then the load of beets is pulled from the wagon by a team of horses. In another method, the loaded net is lifted from a wagon, or car, by a traveling crane and its contents deposited where desired in the bin. Hydraulic dumping devices are often used to unload railway cars. Several cars are clamped on a platform and discharged at one time by tilting them.

About 50 pounds of beets are removed from each load, so that the allowance for tare may be estimated. These beets are weighed, then brushed and washed free from soil and rootlets, and parts of the necks that are carelessly left in topping are removed. After cleaning, the beets are dried by wiping or by drainage and are again weighed. A small arbitrary allowance is often made for the water that is absorbed by the beets or that adheres to them. The loss of weight is used in calculating the deduction to be made for tare. The factory chemist usually has charge of the tare room.

10. Storage of Beets.—As the factories are active long after the proper time for harvesting the crop, large quantities of beets must be stored for use during this period. The method of storing depends on local conditions, the climate, and the factory facilities.

Covered bins, supplemented by storage in the open yard, serve the purpose in California, where the climate is very mild. The piles of beets should not be very high and they should be protected from the sun by a light covering. Great care is required to avoid loss through the heating of the beets in the bins or the piles. Covered bins are also used in other parts of the United States.

Where beets are to be stored for only a short time, they may be collected in large heaps and then protected with a light covering of soil. The storage place should always be well drained to prevent rotting. In parts of the United States where the winters are cold, the beets are often stored in large piles with no covering. The outer layers freeze, but the frost does not penetrate far into the pile. If exposed to alternate freezing and thawing weather, the outer layers rapidly deteriorate. American experience indicates that in cold climates the beets should be protected from thawing rather than from freezing. When beets are stored for a short time in the fields, they are usually protected with a covering of leaves or soil.

In European practice, sheds and silos are employed, while in some localities, storage in the open air is resorted to. The methods of storage in sheds and in the open air do not differ materially from those followed by the American factories. The silos are of various forms, the usual type being arranged as follows: The beets are piled on a well-drained piece of ground and are then covered with a layer of earth. Loose straw covers are sometimes used, but this practice should be avoided on account of the effect of straw in the beet slicers, dulling and stopping up the knives. Ventilators are located quite near one another and extend upwards from the bottom of the pile of beets. The silo is surrounded by a suitable drainage ditch.

11. Hydraulic Transport of Beets From Bins, Etc. to the Factory.—The beets are usually conveyed from the sheds, or bins, to the washing machines by a stream of water flowing in flumes. The floors of the bin slope toward the flume, and the latter is covered with loose planks, on which the roots are piled. As the beets are needed, they are pushed into the flume by the workmen, and at the factory they are lifted from the flume either by a wheel provided with buckets or by a screw conveyer.

12. Composition of the Sugar Beet.—The sugar beet usually contains from 3 to 5 per cent. or more of *marc*, or matter that is insoluble in water. As a rule, only two sugars, *sucrose* and *raffinose*, are present in the mature beet, though it may also contain invert sugar, of which traces are always found in the immature root. Invert sugar is of no value to the sugar manufacturer.

The amount of sucrose present varies greatly with the variety of the beet and the soil and climatic conditions. Small beets are usually much richer in sugar than large ones. The average sugar content of beets produced for the American factories will probably approximate 14 per cent. Beets containing as little as 11 per cent. of sucrose are not desired by the factories, and are seldom purchased except at a reduced price.

In addition to sucrose, raffinose, and chlorophyl, the beet contains a large variety of compounds, namely: *Organic acids*: Oxalic, formic, citric, malonic, succinic, aconitic, tricarballicylic, oxicitric, malic, and tartaric acids. *Nitrogenous bodies*: Betaine, asparagine, glutamine, leucine, legumine, tyrosine, xanthine, guanine, hypoxanthine, adenine, and carnine. *Non-nitrogenous bodies*: Lecithine, pectose, and coniferine. *Mineral constituents*: Salts of potassium, sodium, rubidium, vanadium, calcium, magnesium, iron, and manganese.

The bases are combined with hydrochloric, sulphuric, nitric, phosphoric, and silicic acids and the organic acids named in the preceding list.

The beet also contains the non-nitrogenous bodies, lecithine and pectose. The latter is converted into soluble pectine by the pectase, which is also present. The sugars in the beet are principally sucrose and invert sugar. Raffinose is present in small quantities and accumulates in the molasses. This sugar forms needle-shaped crystals and is not injurious in the

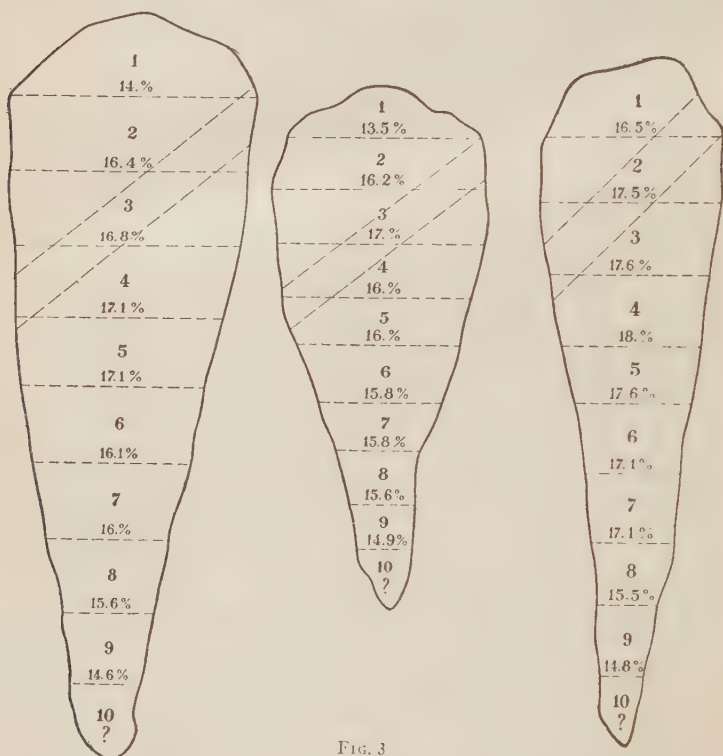


FIG. 3

manufacture. The distribution of the sugar in the beet, according to Slassky (Bul. Assoc. des Chimistes de France, **12**, 277), is shown in Fig. 3.

13. In the purification of the juice, many of these substances are removed by precipitation as lime compounds. Others are carried down mechanically by the precipitates, and

the albuminoids are removed partly by coagulation and partly by precipitation. The precipitant used in the manufacture is lime, the excess of which is converted into the carbonate, which itself acts mechanically in carrying down the suspended impurities of the juice. A part of the lime forms soluble salts, which are troublesome at all subsequent stages of manufacture. The sodium and potassium salts are not removed in the purification of the beet juice, but remain and give it an alkaline reaction after all the lime has been saturated with carbonic acid.

METHODS OF MANUFACTURE

14. Synopsis of Manufacture.—The various stages of the manufacture of beet sugar may be outlined as follows: (1) Washing the beets; (2) weighing the beets; (3) slicing the beets; (4) extracting the juice by the diffusion process; (5) straining the juice; (6) purification of the juice; (7) bleaching the juice; (8) concentration of the juice to a sirup; (9) crystallization of the sugar—(a) in the vacuum pan and (b) supplemental crystallization with motion; (10) curing the sugar; (11) molasses processes, or processes for the recovery of the sugar in the molasses—(a) osmose processes and (b) saccharate processes; (12) utilization of by-products—(a) pulps, (b) molasses, and (c) distillery and other residues from the utilization of the molasses.

PROCESSES AND MACHINERY

15. Washing the Beets.—Part of the washing of the beets is already done by the warm condenser water in the flumes and part of the separation of the beets from stones is accomplished by stone catchers, which are built into the flumes a short distance before the elevating devices.

Trash catchers are attached and built into the flumes and serve to remove beet leaves, beet tops, floating weeds, straw, etc., from the beets. The final washing is done in washing machines built for this purpose.

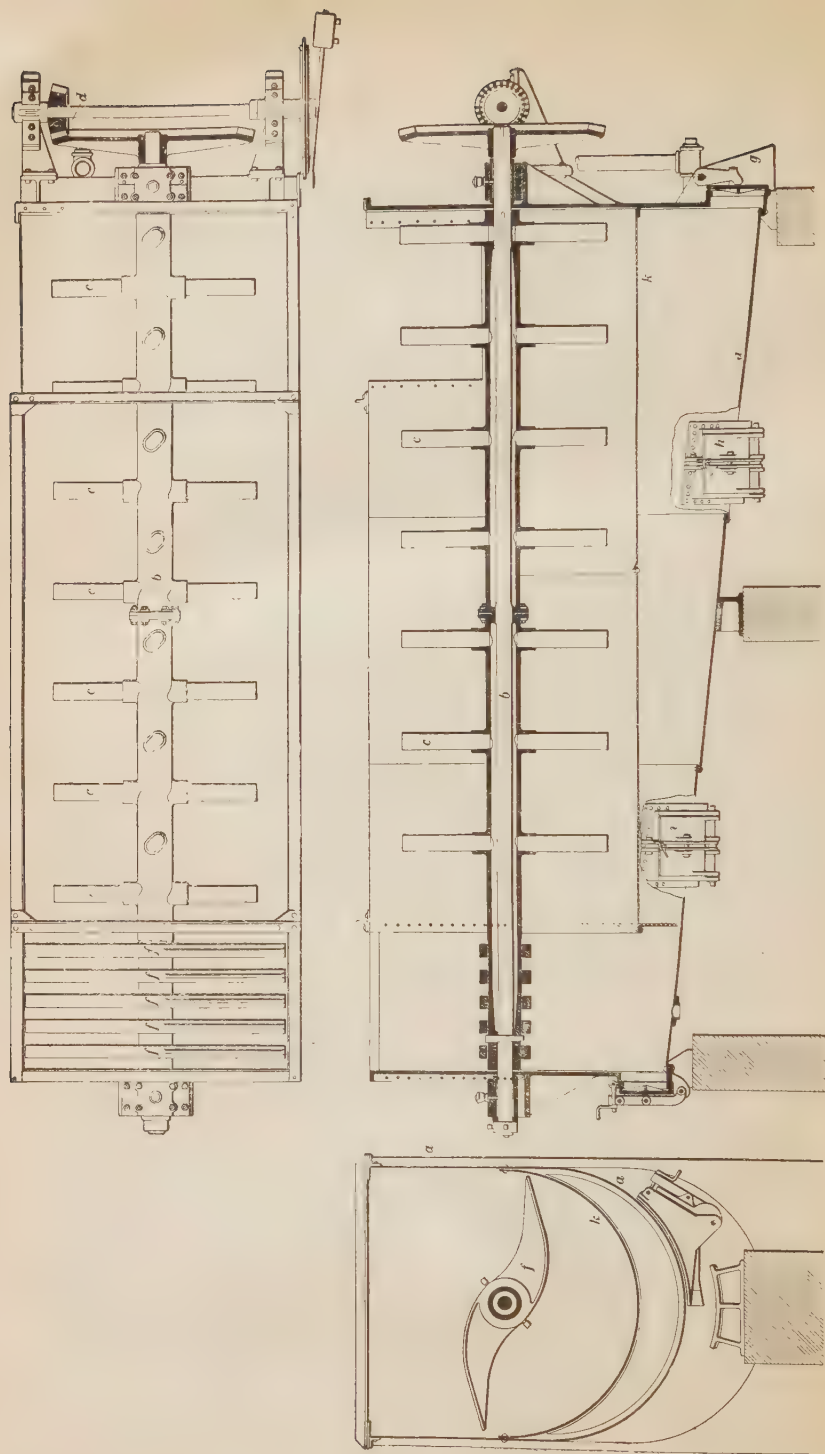


FIG 4

In Fig. 4 is shown a plan view and also a cross-section and a longitudinal section of a beet washer made by E. H. Dyer and Company. The washer consists of a shell, within which is a hollow metal shaft *b*, carrying arms *c* that agitate the beets in the water. The beets conveyed from the beds and sheds through a flume containing running water are raised by means of an elevator wheel and are thrown on an apron, which drops them into the washer having a driving gear *d* and *e* at its end. The beets are thrown out of the washer at the opposite side by the arms *f* on to a small conveyer, or into what is called a *trash catcher*, that is, a sheet-metal box with a screw for conveying the beets from the washer to the elevator. This device delivers them to an automatic scale, which registers their weight and drops them directly into the cutter. Referring again to Fig. 4, *g*, *h*, and *i* are doors in the end and sides for cleaning out the dirt, sand, stones, etc. that separate from the beets and accumulate in the space between the perforated false bottom *k* and the shell *a*.

16. Weighing the Beets.—It is customary, especially in Europe, to weigh the beets immediately before slicing them. Preliminary to weighing, the beets are either passed over rotary brushes or transported a short distance on a shaking conveyer, to complete the drainage of the wash water and to remove stones and earth that may still adhere.

Automatic scales are very generally used in Germany and the United States, the Chronos scale, of German manufacture, being usually employed. The filling, weighing, and discharging of this scale are automatic. Numerous semiautomatic weighing devices are also used. The French frequently employ a scale on which the beets are weighed in a car; an automatic stop arrangement prevents the car from leaving the scale until the load is complete.

The automatic weighing of the beets is desirable where the beets are taxed according to their weight.

17. Slicing the Beets.—At the present time, the diffusion process has replaced all others in the extraction of the juice from the beet. The first step in this process, after the

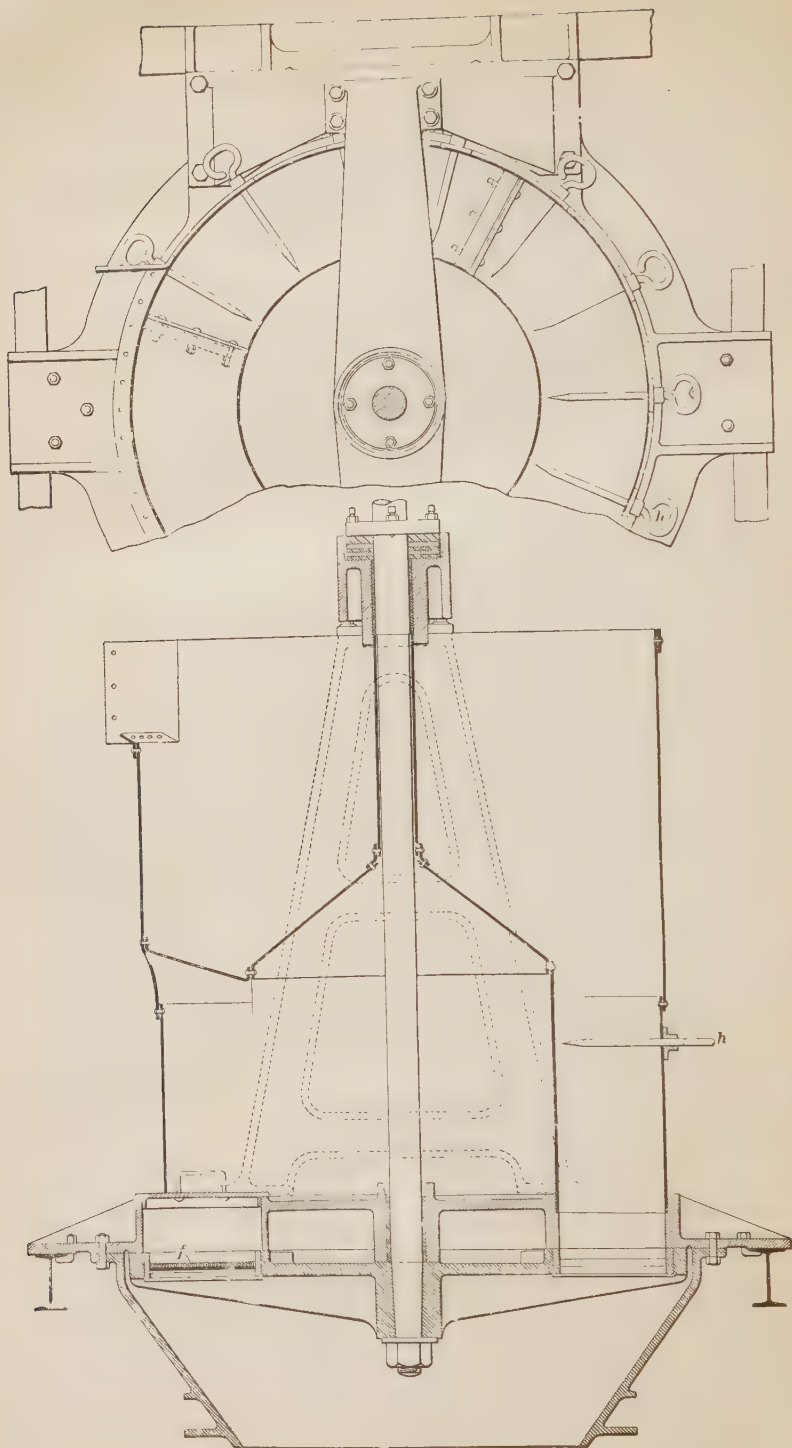


FIG. 5

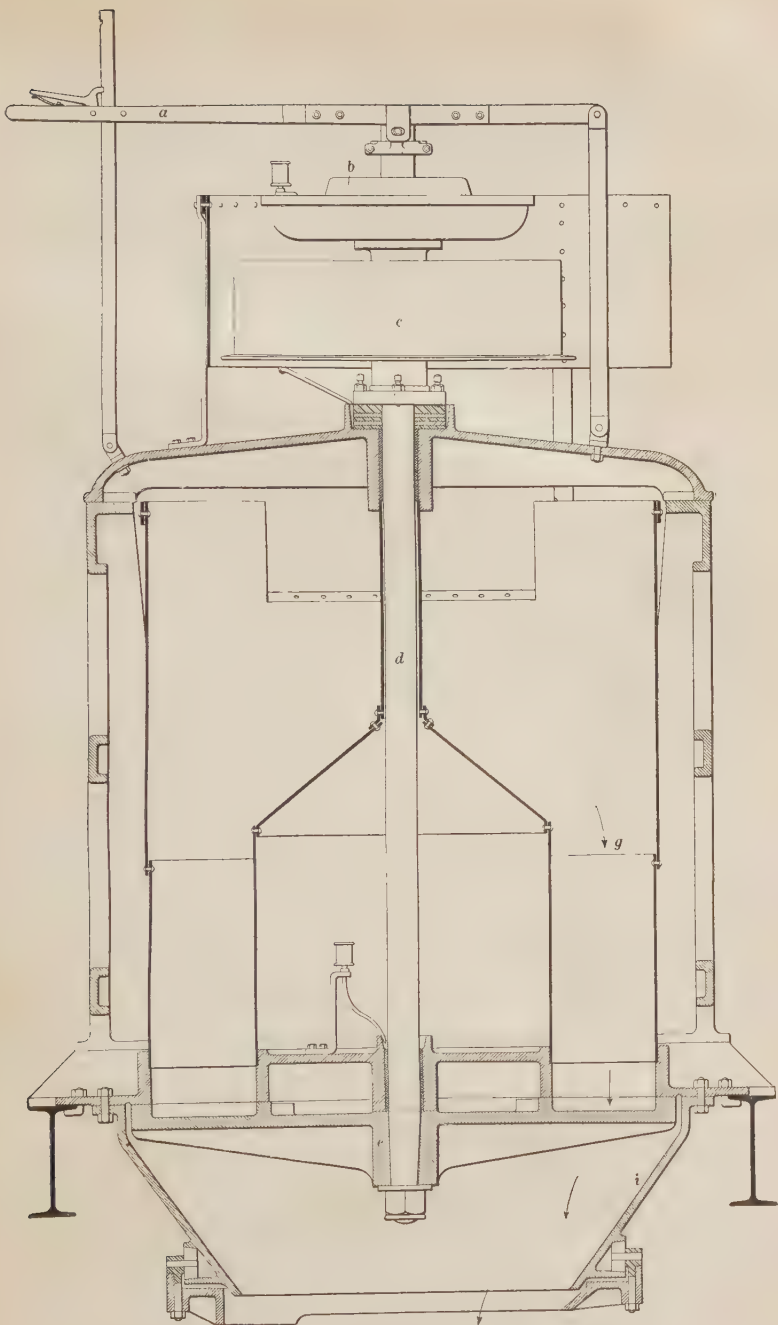


FIG. 6

beets have been thoroughly cleaned, is to cut the roots into thin slices termed *cossettes*, and it is of great importance that these be of uniform thickness. A machine in common use for this purpose is shown in Figs. 5 and 6. This machine is made by E. H. Dyer and Company.

In the figures, *a* represents the lever for throwing the clutch *b* in and out of action, and *c*, the driving pulley that operates the shaft *d* carrying the circular frame *e*, which holds the knives *f*. At *h* are shown pins used to keep the weight of beets off the knives, so that when dull, they may be replaced by sharp ones without unloading the cutter. The beets fall into the cutter and, following the course of the arrows *g*, are cut by the knives *f* into **V**-shaped slices, which are usually $\frac{1}{8}$ inch thick and $\frac{5}{8}$ inch wide. These slices, or *cossettes*, fall through the hopper *i* into a chute, which conveys them to the diffusion-battery cells on the floor below. Some factories use knives that cut a rectangular-shaped cossette, but most of the modern factories prefer the **V** shape, as in the rectangular shape the walls are much thicker and a higher temperature is required to extract the sugar from them, with the ever-present danger of retarding the circulation of the juice through the *cossettes* by softening or matting them by excessive heat.

Another form of beet cutters is the *drum* cutter, or *Maguin* cutter. The knives in this case are inserted in a drum revolving around a horizontal axis. The cutting is done inside of the drum and the beets are introduced into this drum in a wedge-shaped space, so that a pressure of the beets against the knives is obtained. The knives are designated as *rib-roof* knives.

18. Many factories install two cutters for each battery and are thus able to send an uninterrupted stream of *cossettes* to the battery, as a reserve cutter is always ready for use when the knives of one become dull or broken or in case a cutter for any reason gets out of order. Much trouble and delay are caused by stones, iron, etc. being carried along with the beets and breaking the knives; but this difficulty is partly overcome by *stone catchers* of various designs. The most successful of these are placed in the bottom of the

washers, or trash catchers, and are simply sheet-iron boxes filled with water, through which the beets flow on their way to the elevator, the stones falling to the bottom and being removed at regular intervals through a door in the side. Another type consists of a large iron basket with a perforated bottom. The basket is placed under the beet flume, the top of it being on a level with the bottom of the flume. As in the other case, the beets float on over this basket and the stones drop into it.

EXTRACTION OF THE JUICE

19. Theory of the Diffusion Process.—The juice was formerly extracted from the beet by means of hydraulic presses. This method, however, has been entirely superseded by the **diffusion process**. This process received its first practical application in Austria.

The diffusion process depends, in theory, on the property possessed by a class of substances, called *crystalloids*, of passing from one solution into a contiguous solution, through a porous membrane that separates the two liquids. This action is termed *osmosis*, or *dialysis*. Under the same conditions, certain other bodies, called *colloids*, pass through the membrane, but slowly, if at all. The crystalloids of the beet, of which sugar is the principal one, include most of its mineral salts and salts of the organic acids. Certain albumins are among the colloid bodies of the sugar beet. Crystalloids are substances that may be readily crystallized, and the colloids, on the contrary, are not usually crystallizable. The rate of the dialysis increases as the temperature of the solutions increase.

20. In the application of osmosis, in the extraction of the sugar from the beet, the walls of the plant cells themselves supply the membranes. The beets are cut into very thin slices, and these are then bathed in water. The crystalloids pass through the cell membrane during the interchange of the liquids within and outside the cells.

In slicing the beets, as described previously, many of the plant cells are necessarily ruptured, and their contents mingle

with the water with which the cossettes are bathed in the diffusion process. This process is, therefore, both one of osmosis and one of maceration. Sharp knives are always used in slicing the beets, so as to avoid rupturing more cells than is actually necessary. The cell membrane consists largely of cellulose and pectin bodies, which are rendered soft and flabby at certain temperatures. The pectin bodies also become somewhat soluble in water on heating; hence, great care must be taken in the diffusion process to have temperature conditions as nearly perfect as possible.

It should be observed that ideal conditions cannot be attained in the practical application of the diffusion process and that, together with the crystalloids, many of the colloids are extracted.

21. Diffusion Battery.—The apparatus used in the practical application of the diffusion process consists of a series of vertical iron vessels that are provided with suitable doors for charging them with beet cossettes and discharging the exhausted pulp, and also with piping and connections for the circulation of water, juice, and steam. These vessels may be arranged in a single or a double straight line, or in a circle. The series of vessels is termed a **diffusion battery**, and the individual vessels, *diffusers*, or *cells*.

A conveniently arranged circular diffusion battery, manufactured by the Walburn-Swenson Company, is shown in Fig. 7. The choice of the type of battery, whether to be arranged in a line or in a circle, depends on the general scheme of the factory and the preferences of the builder. The various arrangements mentioned are equally effective in extracting the sugar. Double-line batteries are usually preferred, as they do not require a very high building and may be readily enlarged to two single-line batteries. The usual number of diffusers forming a battery is twelve.

22. The battery illustrated in Fig. 7 consists of fourteen cells, C_1 to C_{14} , each of which has at the top a feed-opening that may be securely closed with a swinging cover. Each of these covers has a cock Y , which serves as an air vent

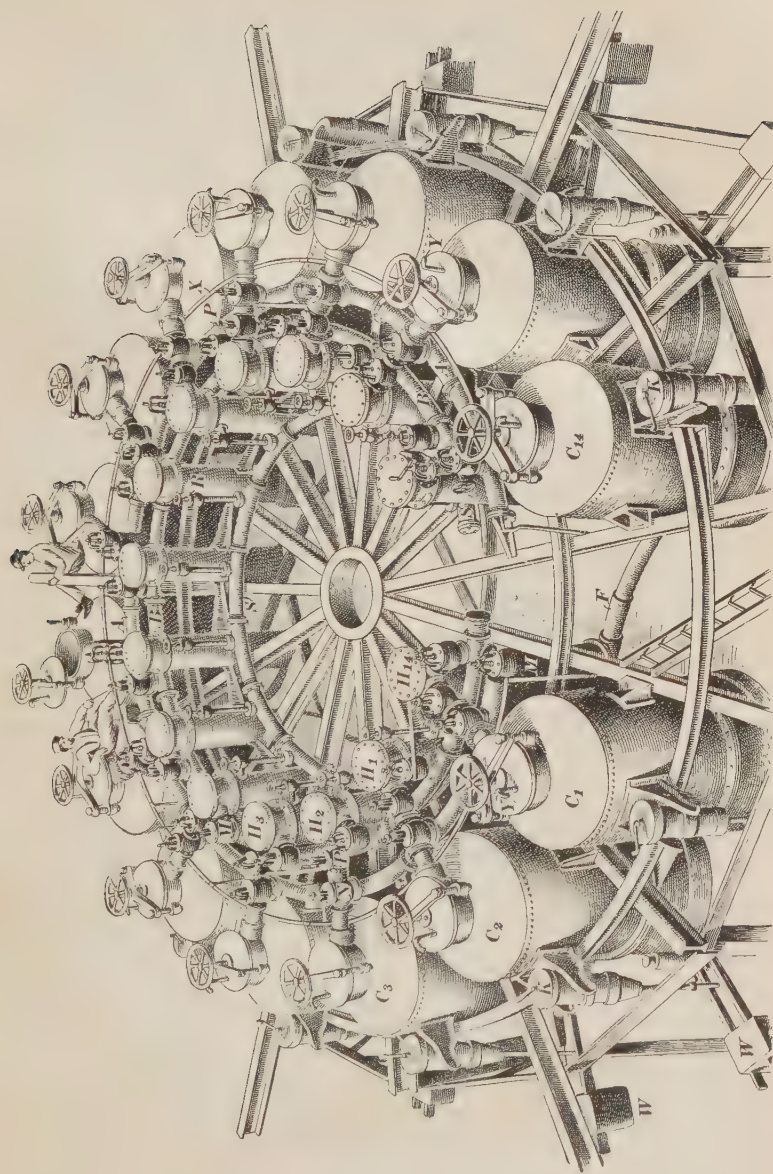


FIG. 7

and as a means of ascertaining when the diffuser is completely filled with juice. At the bottom of each cell is a hinged door for discharging the exhausted cossettes. This door is opened and closed by means of a hydraulic cylinder *K* and a balance weight *W*. Water is supplied the cylinder through the pipe *X*. Each diffuser has a juice heater, or calorisor, *H*₁, *H*₂, etc. and is connected with the bottom of this device by the pipe *F* (shown in the figure only at cell *C*₁₄). The upper part of the heater connects through the valve *M*, and the upper pipe lines connect with the top of the next diffuser of the series. The heaters receive steam through the pipe *S* and the valves *R*. Instead of tube or coil heaters, steam injectors are frequently used. The pipes *A* and *B* are, respectively, the water and juice lines. The cross-valve *N* serves to connect the first cell of the series with the water-line *A*.

In cells with side-door discharges, the bottom of the cell is covered with a perforated sieve on which the cossettes are resting and through which the juice is circulating. In cells with bottom-door discharge, the conical lower part of the cell and the bottom door itself are covered with such perforated sieves. The combined area of the holes in these sieves should be four to six times the area of the circulating pipes of the battery.

In operating the battery, each cell successively becomes the first of the series. Three cells are usually disconnected from the others. One of these three cells is being charged with fresh cossettes, the second is empty and ready to be charged, and the third is being emptied of exhausted pulp. In a few cases a compressed-air pipe, with valves at each diffuser, is provided to transfer the water from the cell containing exhausted pulp to the next diffuser, before removing the pulp. In this arrangement, air is used to force the juice through the series of vessels and the juice from the last diffuser into the measuring tank.

23. Diffusion Process.—With the assistance of the outline of the theory of the process and the illustration of

the machinery, the following description of the diffusion process itself will be readily understood:

In beginning the work, a diffuser is filled with beet cossettes, and warm water is then passed into it through the pipe at the bottom, while the air and gases are vented by means of the cock in the cover. While the first diffuser is being filled with water, the second is receiving cossettes, and so on. When the first diffuser is filled with water, or, as it may now be termed, *juice*, the direction of the current in this diffuser is reversed by a proper manipulation of the valves. The juice is passed up through the first heater into and downwards through the heater of the second diffuser; it enters the latter at the bottom and passes upwards as before.

There are two objects in admitting the juice, or water, at the bottom of the diffuser: (1) the juice passes through two heaters, thus exposing it to a large heating surface, and (2) on entering at the bottom of the cell, the juice drives the air and gases ahead of it and distributes itself uniformly throughout the cossettes. When the second diffuser is filled with juice, the direction of the current is changed in it, as in the previous vessel. When nine cells of a battery made up of twelve cells have been filled as described, a measured quantity of juice is drawn from the last, the circulation of the liquid being maintained by passing water into the first diffuser of the series. During these manipulations, the temperature of the juice in the various diffusers is regulated to meet certain conditions that will be described later.

After drawing a quantity of juice from the ninth cell into a measuring tank, the first diffuser is cut out of the series and opened. The pulp, or exhausted cossettes, that this diffuser contains is then discharged from it. If the battery is composed of more than twelve diffusers, all but three are filled before drawing a charge of juice or discharging the pulp from a diffuser. After the first cell is cut out, the second becomes the first of the series and is the next to be emptied. From this time on when a cell is filled with fresh cossettes and liquor, a charge of juice is drawn from it and the first diffuser is emptied. As just explained, the juice is always

circulating through all but three of the diffusers; hence, the cossettes are in contact with the diffusion juice of various densities for about 1 hour and 20 minutes, or longer. The concentration of the juice increases from diffuser to diffuser; that of the first vessel contains only a small fraction of a per cent. of sucrose, and the juice of the last cell has a density of about four-fifths of that of the normal beet juice.

24. The exhausted cossettes are removed from the factory by means of a screw conveyer or a drag, or they are flushed from it with water. In later years pulp pumps are used almost exclusively for the transportation of the pulp from the pit underneath the battery to pulp silos or pulp dryers, and since these pumps use a certain amount of water, the use of air at the last cell of the battery is almost entirely abandoned. The further treatment of this by-product will be considered later. Under favorable working conditions, this residue contains from .15 to .25 per cent. of sucrose, in terms of the weight of the beets.

There is a small loss of sucrose in the waste waters that come from the battery, and there is always some water or very thin juice left in the first diffuser after drawing the measured quantity of juice from the last vessel. This thin juice contains only a trace of sucrose.

25. From the foregoing description of the operation of a diffusion battery, it will be seen that the theoretical conditions of the process are fairly well complied with in its practical application. Thus, the thin slices of the beets, which expose the cell membranes of the plant, serve to separate the beet juice from the water used in extracting the cossettes. The slices remain in contact with the liquid for more than an hour, or long enough for the principal crystalloid—sucrose—to pass through the membrane. As will be shown further on, the temperature of the battery is regulated so as to secure rapid dialysis without destroying the cell membrane. However, notwithstanding all precautions, the juice obtained is quite impure, considering everything except sucrose as impurities, because

many plant cells are ruptured by the slicer and many crystalloids besides sucrose are extracted.

As previously stated, a measured quantity of juice is drawn from the last diffuser. This juice is conducted to the next station of the factory for purification. The quantity of diffusion juice to be drawn depends on the quality of the beets and the amount of dilution of juice that manufacturing conditions will permit. Under favorable conditions, the density of the diffusion juice should be about four-fifths of that of the normal juice. To obtain juice of this density, and at the same time exhaust the cosettes of sugar sufficiently, from 109 to 113 liters of juice should be drawn per 100 kilograms of beets. This volume of juice is varied according to the richness of the beets, but is fairly constant during the greater part of the manufacturing season. This is due to a decrease in the volume of the normal juice of the beet as the latter increases in maturity. The juice is usually measured in a rectangular or circular tank, the batteryman recording the volume and temperature of the juice drawn on a blank form. The record of the volume is often kept automatically by the Horsin-Déon or some other recording device.

26. Temperature Conditions.—The control of the temperature at which the diffusion is conducted is of great importance. If the temperature is too high, the cell membranes of the beets break down, or are *cooked*, and the pectic bodies pass into solution in the juice. As the dialysis is rapid at high temperatures and sluggish with cold solutions, the diffusion is conducted at as high a temperature as is practicable without affecting the stability of the cell membranes. Experience has demonstrated that the maximum temperature should not exceed 85° C. If the temperature is too high, the exhausted cosettes cannot be properly pressed for use as cattle food, and they may even pack in the diffusers and thus slow down the circulation of the battery and render their discharge from the battery very difficult. Perfectly sound beets withstand a higher battery temperature than those which have been stored a long time or have been frozen. M. François Dupont,

President of the French Association of Sugar Chemists, recommends the following range of temperatures, in degrees centigrade, for the active diffusers:

Diffuser No. 1	2	(3, 4, 5, 6, 7, 8)	9	10
Temperature 40°	60°	76° to 85°	65° to 70°	40° to 50°

Diffuser No. 1 contains the exhausted pulp and No. 10, the fresh cossettes. The maximum temperature should be lower when treating beets of poor quality.

With healthy beets, it is desirable to keep the temperature as high and as close toward the head of the battery as is possible in order to coagulate albuminous substances inside of the plant cells and thus prevent them from entering the diffusion juice.

27. Remarks on the Conduct of the Diffusion.—In the event of a long stoppage of the manufacture, it is necessary to empty the diffusion battery. To accomplish this with little loss of sucrose, the usual procedure is as follows: Two charges of juice are drawn from the last diffuser, and the pulp is then discharged from the first vessel; two more charges are drawn, and the pulp is then discharged from the second diffuser. This operation is continued, drawing two charges of juice from the last cell and each time discharging a diffuser. Not too many cells should be emptied because it is easier to keep the necessary temperature with six or seven cells than with fewer cells. The process of *sweetening off the battery* may be considered as finished when the juice drawn from the last cell has .75 to 1.00 per cent. of sugar. Then the cells may be emptied one after the other as fast as the pump will take the pulp away.

Should the supply of beets be insufficient for operating the plant at its full capacity, the rate of slicing should be reduced and the battery should be run at a comparatively low temperature. For satisfactory work, the cossettes should be smoothly cut with sharp knives and should be thin but not too thin. The work should be conducted rapidly and with regularity, and the water supplied to the battery should be of good quality and free from bicarbonates, sulphate of calcium, or other sub-

stances that would deposit on the surfaces of the heaters, etc. It is common practice to conduct the work at the rate of 200 diffusers of cossettes per 24 hours.

It is of utmost importance for good diffusion work, which is the fundamental condition for good work all the way through the house, that the work goes on regularly without interruptions and as fast as possible. The faster the juices move through the house, the healthier they are as a general rule. First-class cossettes are indispensable for good work, but the knives do not stay sharp all the time—sand, stones, straw, etc. may interfere and poor cossettes will occasionally come into the cells, notwithstanding all care and vigilance. Sprouted, woody beets, and, later in the campaign, fermented beets will have to be handled and will be the cause of poorer cossettes. If such poorer cossettes are treated at high temperature in the battery they get soft and mushy and stop up the sieves in the diffusion cells and slow down the circulation of the battery.

To relieve the pressure of pulp and cossettes from the sieves, chains inside of the battery cells are now generally adopted in American beet sugar factories. They are arranged in crosses or several sets of crosses, or basket, at different distances from the bottom sieves and may be considered as one of the most effective protections against slowing down of the battery. Since these chains have been adopted, it has been possible to increase the water pressure on the battery from around 20 pounds to almost 40 pounds and thus increase essentially its capacity.

PURIFICATION OF THE JUICE

28. Straining, or Depulping, the Juice.—As it is drawn from the diffusion battery, the juice contains large quantities of minute fragments of beet pulp. These must be removed prior to the carbonation process, since the pectin bodies would be decomposed and, at least in part, be rendered soluble. The depulping, or straining, is accomplished either between the diffusion battery and the measuring tank or after the juice leaves the latter.

A common type of strainer consists simply of a metal basket with fine perforations, through which the juice is caused to flow. This basket is placed in a suitable receptacle and is protected from the air. It is removed from time to time for cleaning. In another type of depulper, finely perforated brass sheets are formed into pockets similar to the cloth bags of the mechanical juice fillers. The juice passes, under low pressure, from the outside of the pocket to the inside and leaves the pulp on the strainer, from which it falls when much of it has accumulated. The pockets are placed in an iron box and are so arranged that the clean juice may be led off from the inside of them and the pulp removed as required.

29. Reheating the Juice.—As the juice leaves the diffusion battery, its temperature is from 40° to 50° C. This low temperature was formerly considered desirable at the beginning of the carbonation, but the present practice is to heat the juice to about 90° C. The object of this heating, aside from the economy of fuel, is to coagulate a part of the albuminoid matters. The heating is accomplished by passing the juice through calorisors that are heated either by a part of the vapors from the first or second body of the multiple effect or by those from the last pan on their way to the condenser. These latter vapors are, however, not very effective on account of the small difference in temperature.

The reheating of the juice before the lime is added, is of the utmost importance and is generally practiced. The colder the juices are, the more lime goes into solution which in the following heating before or during the carbonation is precipitated as hot tri-calcium saccharate. It is hard to carbonate in this form and leads to high sugar losses in the limecake.

30. Purification of Juice by the Carbonation Process.—The double-carbonation process is always used in the purification of the beet juice. This process is divided into the *carbonation* and the *saturation*, or *second carbonation*. In the carbonation, a large quantity of lime, usually from 2 to 3 per cent. of the weight of the beets, is added to the juice,

and then all but a small quantity of it is precipitated with carbonic acid. After the removal of the precipitate, sometimes another small quantity of lime—about .5 per cent. of the weight of the beets—is added to the juice, and is then precipitated with carbonic acid, together with that left in the previous operation. In this second operation, all the lime is precipitated; hence the use of the word *saturation*. After a brief discussion of the objects of the liming and carbonating, the carbonation process itself will be described more in detail.

31. Purposes of the Liming and Carbonation.—The lime added to the juice combines with the free acids of the latter and also with those in weak combination; it also forms a soluble saccharate with a part of the sucrose and it precipitates the silica, the pectic bodies, and the albuminoids in part. The lime compounds, with the acids of the juice, consist of both soluble and insoluble salts. The combination of the alkalies, sodium, potassium, and ammonium are broken up and their carbonates are formed. Lime salts are formed in part also from the decomposition products of the glutamine and asparagine. During carbonation, the soluble alkalies are converted into carbonates, as is also the lime. The calcium carbonate entrains and also occludes many of the mechanical impurities and carries them down with itself.

The precipitation of all the lime in the carbonation is avoided, so as to prevent already precipitated impurities from being broken up. After these compounds have been removed by filtration, in the second part of the process, or the saturation, all the lime is removed by precipitation.

The carbonation process employed for beets corresponds to the defecation process used for sugar cane, and its purpose is to remove, so far as is possible, all the non-sucrose from the juice, preparatory to the crystallization of the sugar. The fundamental difference, however, between the lime purification in beet and in cane juices is the absence of glucose in the first and its presence in the latter juices. Glucose is rapidly decomposed by lime at higher temperatures, forming smeary, gelatinous, and dark-colored decomposition—products of the glucose

with lime which partly remain in solution and render the following boiling almost impossible, besides discoloring the juices. Of 3 parts of glucose, if treated with lime at high temperature, enough high molecular organic acids are formed to combine with 1 part of lime and keep it in solution. Therefore, beet juices that are free from glucose can be treated with lime at high temperature and derive all the benefits of the purification at high temperature, while cane juices, if treated with lime, have to be kept at low temperature, say below 40° to 50° C.

32. Carbonation Process.—After reheating the juice to 65° or 75° C., a quantity of quicklime, in lumps, equal to from 2 to 3 per cent. of the weight of the beets from which the juice was derived, is slaked in the juice; or, an equivalent quantity of milk of lime may be used, in which case the temperature of the juice may reach 85° C. In former years the lump quicklime was generally used in Germany, while now slaked lime is used there and in other European countries and in the United States. If the factory is equipped for the recovery of the sugar from the molasses by a lime saccharate process, the saccharate is used in liming the juice.

After liming the juice, carbonic-acid gas is forced into it until the alkalinity is reduced to the equivalent of 1 to 1.6 grams of calcium oxide per liter of juice, as indicated by phenolphthalein. The attendant judges this point by examining a test sample held in a spoon. The precipitate at this time appears granular and the liquid yellow. This test is frequently verified by rapid chemical analysis, in which a standardized sulphuric acid, with phenolphthalein as an indicator, is used. Other indicators, for example, coralline, may be employed, but the apparent alkalinity varies somewhat with different indicators. The experienced workman learns to note the approaching close of the operation by the appearance of the precipitate and the sound of the escaping bubbles of gas. As, under favorable conditions, the carbonation lasts only about 10 or 15 minutes, the tests must be made very rapidly. Excessive carbonation, resulting in impure juices, must be avoided. During the carbonation, large quantities of foam are formed.

This may be beaten down by jets of steam or by the application of tallow or oil in small quantities. Very deep tanks are often employed so as to avoid the use of steam or oil. These tanks are frequently more than 20 feet in depth.

When the test indicates that the juice has been sufficiently carbonated, the juice is quickly heated to 90° C. and filtered through process.

33. Saturation.—The **second carbonation**, or **saturation**, is conducted as follows: To the filtered juice from the carbonation, sometimes a small amount of lime, say 2 to 4 pounds per 100 gallons, is added. Carbonic-acid gas is again forced into the juice until all the lime present that is precipitable by this reagent has been saturated. The juice should remain alkaline after this second carbonation, owing to the presence of alkalies other than lime. The end of the reaction may be ascertained by a titration with a standardized sulphuric-acid solution, or less accurately by means of phenolphthalein test papers. The alkalinity that should remain varies with the beets, but is usually equivalent to .02 gram of lime per liter of juice. When the saturation is completed, the juice is heated to boiling and is then filtered through filter presses. If this carbonation is not carried far enough, still leaving an excess of lime in the juice, difficulty may be experienced in boiling the liquors in the vacuum pan. The evaporation will progress slowly or not at all.

The process has been described as it is usually applied. It is sometimes modified by using other quantities of lime or other temperatures, or by carrying the carbonation somewhat further. The filter-press work is usually a guide in conducting the carbonation. If the press cake is very white and chalky, more lime than is necessary has been used. With insufficient lime, the filtration is sluggish and the press cake is pasty. The proper quantity of lime yields a grayish-colored cake, which is firm and readily washed.

34. Production of Lime and Carbonic Acid.—The carbonic acid for the precipitation of the lime and the lime itself are obtained by calcining limestone in a special type of

lime kiln. The kilns in former times were conical and were constructed entirely of brick with a lining of firebrick. In the present construction, a large iron casing, the frustum of a cone, is supported on columns and at its lower part is suspended a bottom section that is also the frustum of a cone. These sections are fastened together at the bases, forming an enlarged section of the kiln. The upper section terminates at the top in a feed-opening for coke and limestone and in an emergency chimney. The lower end of the bottom section is open and near the ground, on which is built a conical distributor. The iron casing is lined with firebrick.

In starting the kiln, limestone is piled on the distributor until it closes the lower opening; then kindling is thrown into the kiln at the feed-door, and this is followed by alternate charges of coke and limestone in the proportion of 1 volume of coke to 4 or 5 volumes of stone. The charges should be small, so that the fuel and stone may be well mixed and distributed. The fire is started and is maintained by natural draft through the chimney until the stone begins to decompose and the gas is required in the factory. A large pipe leads from near the top of the kiln to the gas washer and thence to the carbonation tanks.

35. The function of the washer is to cool the carbonic-acid gas and to remove impurities. In one of the usual types, the gas flows in a direction opposite to a stream of water that falls in cascades from shelf to shelf of the washer. About 24 hours after lighting the kiln, carbonic-acid gas may usually be drawn from it through the pipe and washer by means of the gas pump and be forced into the juice in the carbonation tanks. The kiln should supply a gas containing about 30 per cent. of carbonic acid and should be practically free from carbonic oxide.

At intervals of two to four hours, lime is drawn from the kiln, which is frequently charged with stone and coke in the proportions already stated. The type of kiln described is used by most large American factories and is one of the most efficient.

The lime kiln is one of the most important stations in a beet sugar factory and especially in a factory with Steffen's separation. The proper handling of the lime kilns determines the success of the Steffen house work.

36. The first condition for good lime-kiln work is good limestone. The chemical composition of limestone should be as pure as is obtainable. The chemical composition of the limestone, however, does not alone determine the value of the stone for kiln work; the physical structure of the stone is also of importance. The stone must be homogeneous; crystalline limestone falls to powder in the kiln and stops the air circulation. The size of the stone going to the kilns should be 4 to 6 inches in diameter and should be as uniform as possible. The size of the coke should be 2 to 4 inches in diameter. The main thing to be watched in the lime-kiln work should be a free passage for the gases of combustion. To put spalls and splinters of limestone into the kiln together with the regular sizes, is about the worst economy one could try to accomplish and might result in a penalty ten to twenty times as high as the value of those spalls, because these spalls or stone splinters fill the voids left by the bigger stones, stop up the gas circulation, slow down the combustion, cause the fire to burn unevenly, overburn one side of the kiln and underburn the other, slow down the capacity of the kiln and thereby the capacity of the Steffen house, etc.

The amount of coke needed is dependent on the size of the kiln. For a 1,000-ton house with a kiln of 2,500 to 3,000 cubic feet capacity, 10 per cent. of coke of 85 per cent. fixed carbon will be about the right amount of fuel.

The size of the kiln may vary in wide limits. In large kilns a slow-burning process at lower temperature is going on, while in smaller kilns the burning process takes place at a higher temperature in a shorter time.

37. The gas pump is the regulating factor of this burning process. The lime kiln is only the place where the limestone is burned, the burning process is speeded up or delayed by higher or lower speed of the gas pump. 100 parts of lime-

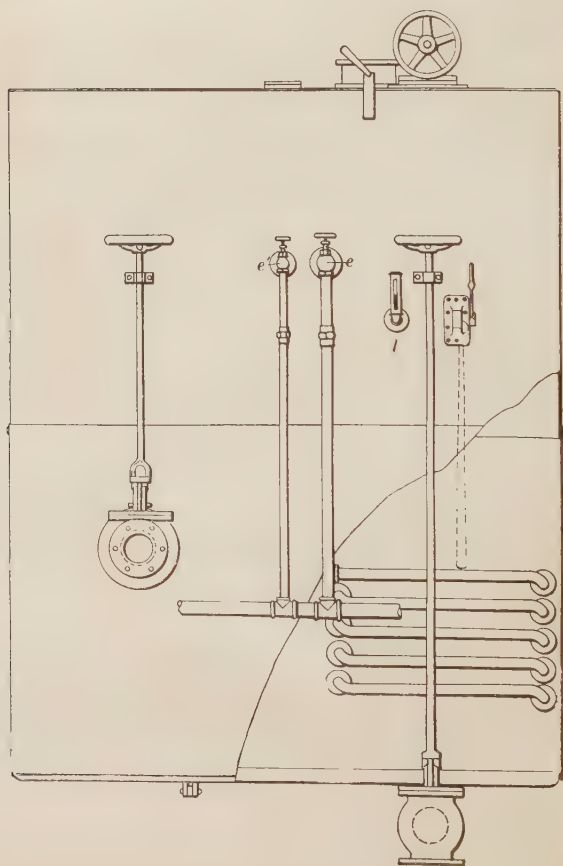
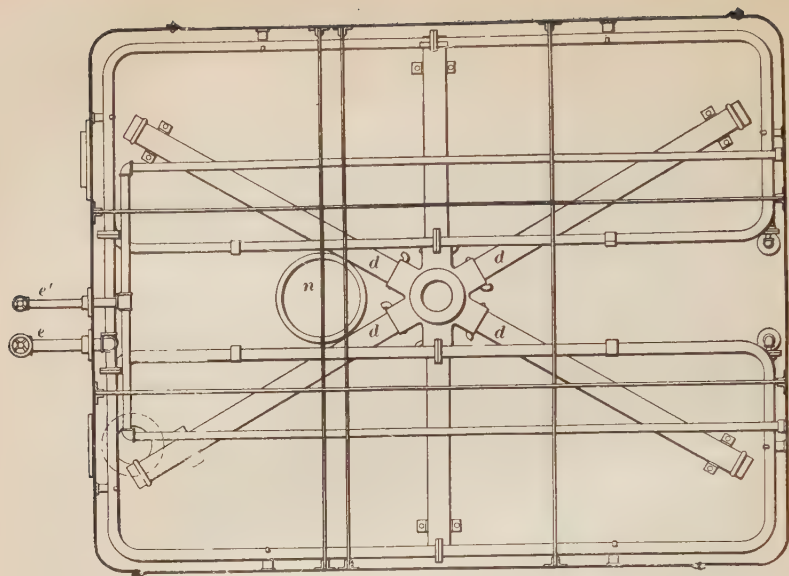


FIG. 8

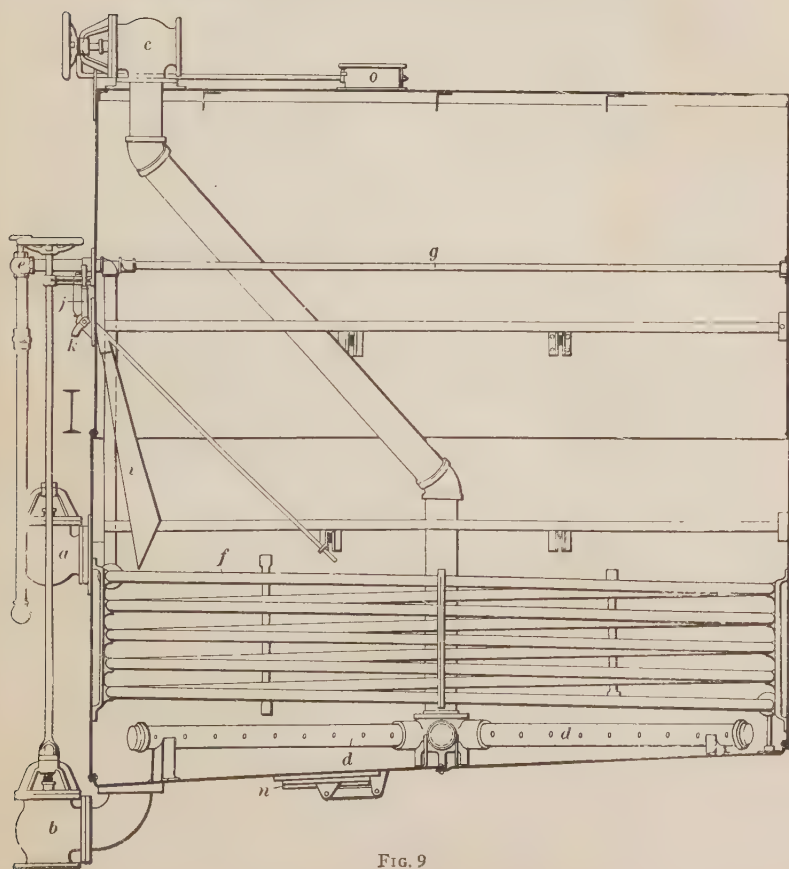
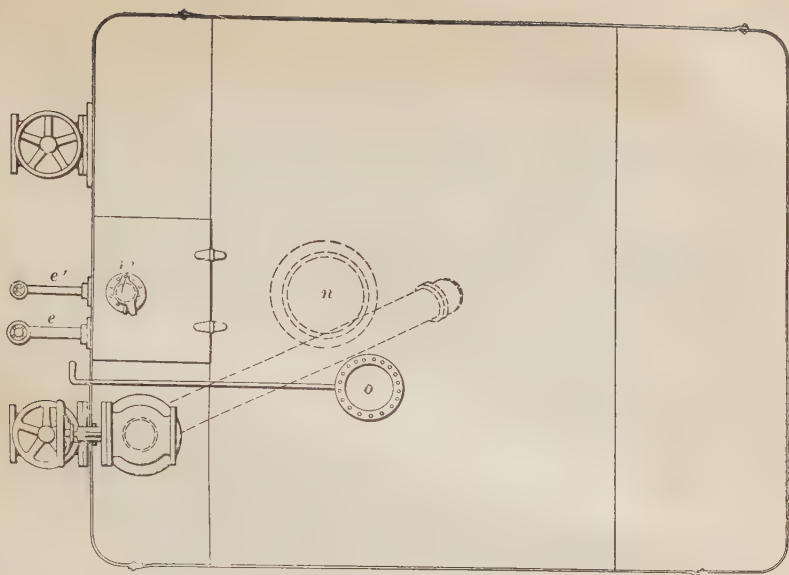


FIG. 9

stone (if chemically pure) give 56 parts of lime and 44 parts of CO_2 . If 10 per cent. of coke with 85 per cent. fixed carbon is used, another 31.2 parts of CO_2 will result from the coke, that is, 100 parts of limestone give 56 parts of CaO and 75.2 parts of CO_2 . To carbonate the same amount of lime, only 44 parts of CO_2 are necessary, or 58.5 per cent. of the CO_2 available. In a good carbonation, about 74 per cent. of the gas is utilized so that only 60 out of 75 available parts of CO_2 are needed, or 20 per cent. of gas is in excess. Therefore, the gas pump has to blow out continuously the excess of gas to the air to burn the necessary lime. Or, the factory could buy 20 per cent. of the necessary lime as burned lime and have enough gas to carbonate it, which, under special freight conditions, might be advisable.

38. Carbonation Tanks.—Figs. 8 and 9 are illustrations of an E. H. Dyer and Company **carbonation tank**, in which *a* represents the inlet valve for the diffusion juice; *b*, the outlet valve for the finished, or carbonated, juice; *c*, the valve for the entrance of the carbon dioxide, which is forced into the juice through the perforated pipes *d*; *e*, *e'*, the steam valves supplying steam for the heating coils *f*, and for the perforated pipe *g*, the latter being used for reducing the foam on the juice; *i*, the sampler for drawing test samples of the juice, which may be obtained without opening the tanks by simply pressing down on the handle *j* and holding a dipper under the spout *k*; *l*, a thermometer; *m*, a sliding cover; *n*, a manhole in the bottom of the tank for cleaning purposes; and *o*, an opening to the vent pipe, which liberates superfluous gases to the outside air through a stack.

The gas-distributing pipes are often so arranged that they may be readily removed from the tank for cleaning. The lime scale that forms on the outer surfaces of these pipes and obstructs the perforations can be easily loosened by heating the pipe in a blacksmith's forge, and the scale that accumulates on the walls of the tank and the heating surfaces can be removed either by scraping or by boiling acidulated water in the vessel.

Before workmen enter a tank, it is always necessary to ascertain whether the tank is free from carbon dioxide and monoxide.

The carbonation and saturation may also be conducted in a continuous carbonator; that is, an apparatus into which limed juice is constantly flowing and from which there is a continuous stream of carbonated juice. Such apparatus, however, is seldom used.

39. Sulphuring.—Sulphurous-acid gas is applied in beet-sugar manufacture, as in the cane-sugar industry, to obtain a white product or to increase the yield of sugar, and the process is termed sulphuring.

Practice differs somewhat as to the stage of the manufacture at which to use the sulphurous acid, but usually it is the filtered juices from the saturation, or second carbonation, that are sulphured.

The juice may be sulphured either with or without again liming it, but usually no lime is added. In the latter case, all the lime is not precipitated in the second carbonation; the carbonating is stopped when the alkalinity is equivalent to about .5 gram of calcium oxide per liter, and the juice is then filter-pressed. From the presses, the juice flows into sulphuring tanks, where sulphurous-acid gas is passed into it until the alkalinity is reduced to the equivalent of about .1 gram of lime per liter. The quantity of lime left from the second carbonation or that added before sulphuring must be sufficient to insure the proper filtration of the juice. After sulphuring, the juice is heated to the boiling point and again filtered. Special filter presses or mechanical filters are used for the filtration. Thick juice is sulphured in nearly all factories.

40. Sulphur Stoves.—There are two general types of sulphur stoves, namely, *closed stoves*, into which air is forced by a pump; and *open stoves*, in which the draft is induced either by an ejector in the chimney or by a pump. The closed stoves deliver the gas under pressure and are used almost exclusively in beet-sugar manufacture, while the second type is used in the cane-sugar industry.

41. The closed stoves are of various forms, but in cross-section they are usually the shape of an inverted **U**. This type of stove is made of thick cast iron, as is also the gas pipe leading from it. At the front of the stove is a door that forms a tight joint when closed and serves for the introduction of sulphur or for cleaning the apparatus. The sulphur is burned in a cast-iron pan. Both the stove and the gas pipe are water-jacketed for the cooling of the gas. This is advisable, because sulphurous acid is decomposed into its elements at high temperatures. Air is forced into the stove by a compressor and is usually delivered at the front of the sulphur pan or under it. The stoves are often provided with baffle plates so as to prevent sublimed sulphur from reaching the gas pipes; they are also often arranged for charging with sulphur while in use.

To operate a closed stove, the pan is filled with sulphur and a piece of red-hot iron or a lighted fragment of burlap bagging saturated with sulphur is dropped into the pan; the door is then closed tightly and the air compressor is started. The quantity of gas produced is regulated by altering the speed of the air compressor.

42. Sulphuring Tanks.—The sulphuring tanks do not differ materially from those used in the carbonation. As there is no frothing of the juice during sulphuring, the tanks may be almost entirely filled with juice. The valves for admitting the gas are made of hard rubber or of a lead composition; if made of some other material the acid would be liable to destroy them quickly.

Instead of using tanks similar to carbonation tanks, a continuous process of sulphuring is often employed. In one form of continuous apparatus, a tank is divided into five compartments by partitions so arranged that the juice passes under the first of these into the second compartment and overflows into the third, and so on. Sulphurous-acid gas is forced into the juice in each compartment through a perforated pipe. There is sufficient travel for the juice to insure thorough sulphuring. The quantity of gas used is regulated by means of the air compressor. The tanks may be of wood and are located prefer-

ably at the top of the factory, to permit the escape of the gas, provided none is absorbed. After sulphuring the juice, it is heated to boiling, preliminary to its filtration.

FILTRATION OF THE JUICE

43. Filter Presses.—The juices from the first and second carbonations and often the sulphured juices are filtered through cloth in filter presses. Formerly, the juice from the first carbonation was separated from the precipitates by decantation. Filter presses are nowadays generally of the plate-and-frame type.

44. In Fig. 10 are shown many details of a *frame press*, and in Fig. 11 an installation of this type of press is illustrated. The press consists of a series of grooved plates and frames that alternate with one another. At *a*, Fig. 10, is a frame and at *b* a plate. The vertical grooves of the plate at their lower ends connect with a horizontal channel and through an opening with a juice cock *j*. The plates and frames have side lugs that rest on beams *c*. At *d* and *e* are shown the head-piece and the tail-piece, respectively. The screw for clamping the plates and frames together is shown at *f*. A sheet-iron pan, or tray, *k* serves to catch the juice that drips from the cloths or leaks from the press. The juice trough *g* has two outlets *h* and *i*, one for filtered juice and the other for wash water. At the lower part of each plate and frame, as shown in Fig. 11, there are two lugs, one on each side. In each of these lugs is an opening, forming, in one side of the press, the juice and precipitate channel, and in the other, the wash-water conduit. The juice channel connects directly with each frame, and the water conduit with the grooves of every alternate plate, that is, with the *water plates*. The cloths in this type of press have no holes in them. The joints between the channel lugs shown in Fig. 11 are made with cloth *stockings*, which have openings in them that correspond with those in the lugs. In the Kroog filter press of this type, these joints are made with rubber rings or gaskets.

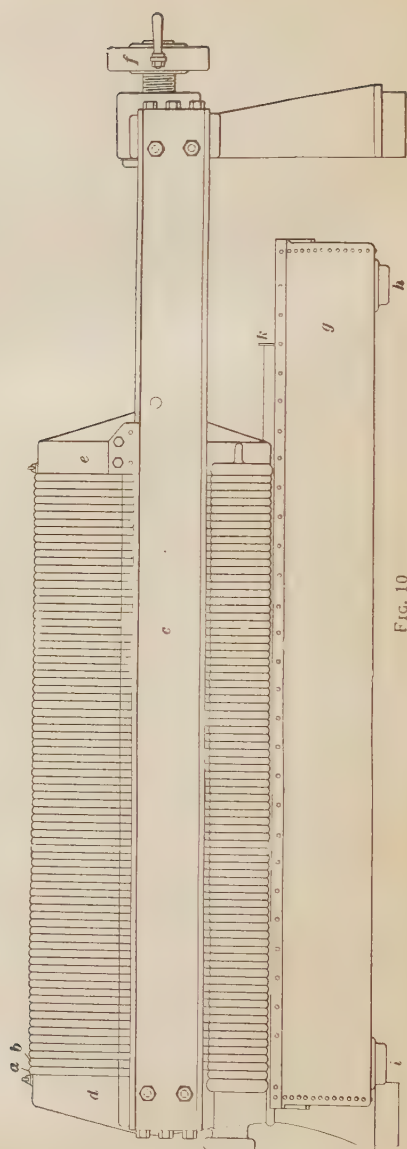
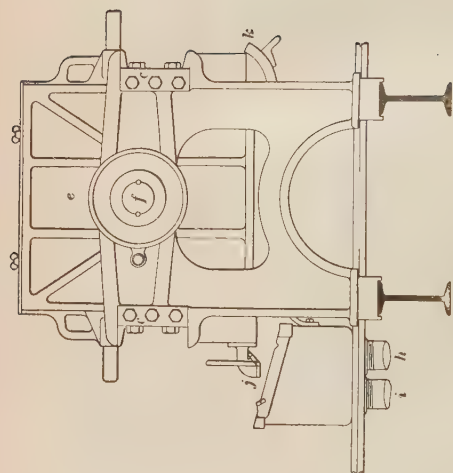


FIG. 10

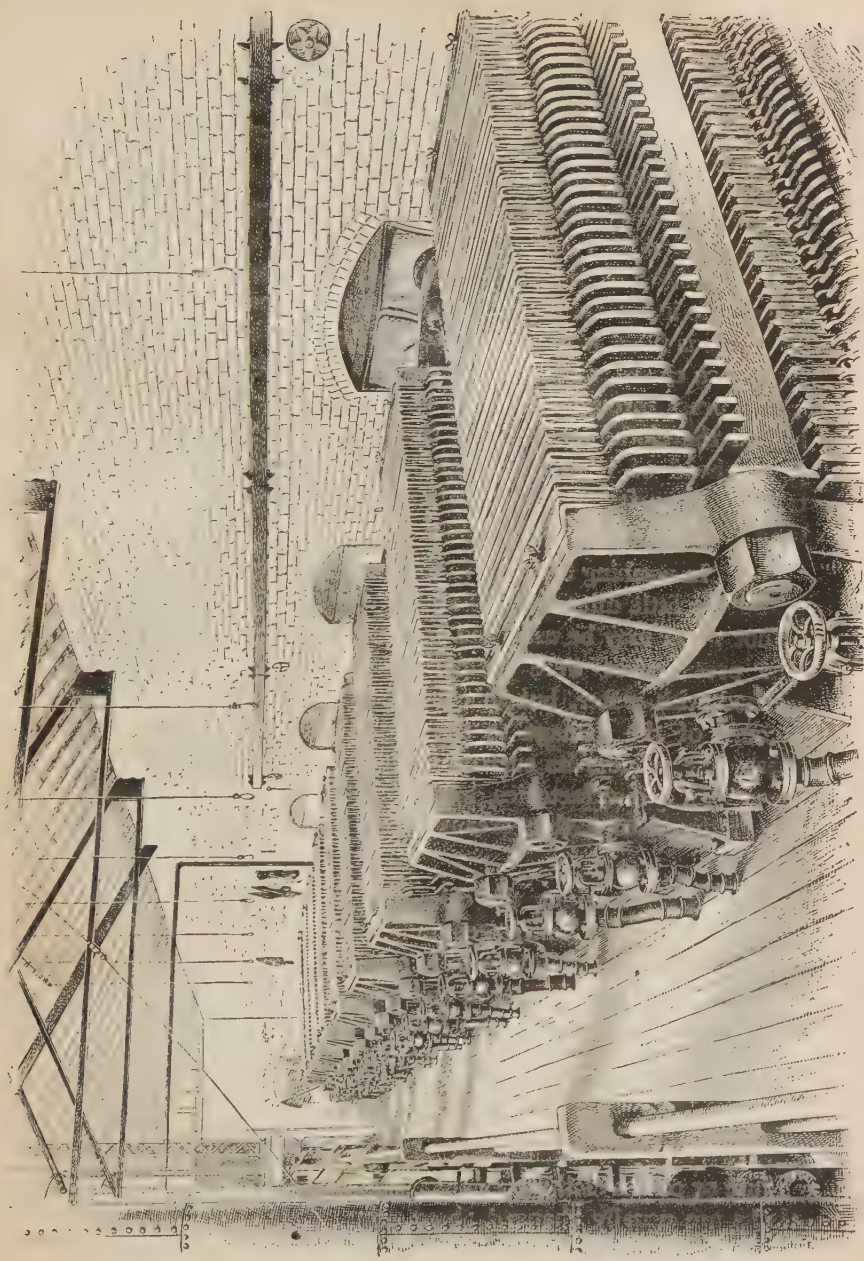


FIG. 11

In *dressing* the press, the cloths are laid over the plates so as to form joints between the plates and the frames, as shown in Fig. 11, and the clamp screw *f*, Fig. 10, is then tightened.

45. Referring to Fig. 11, when the press is in operation, the juice is admitted to it under pump, or monte-jus, pressure through the valve *l* and is distributed through the channel into the frames. The juice filters through the cloth, leaving the precipitate in the frame in the form of a compact cake. When the press is filled with precipitate, the juice cocks on the water plates are closed, as is also the valve *l*, and water is admitted to the press through the valve *m*. The water fills the spaces behind the cloths, passes into the press cake, and drives the juice ahead of it and through the open juice cocks to the trough. The frame presses often are designed to produce a cake about $1\frac{1}{4}$ inches in thickness.

46. In washing the cake in the frame presses, there is comparatively little dilution of the juice. For washing either cold or hot water may be used and apparently with equal efficiency. The press cakes were formerly steamed, instead of being washed with water, but this method has been abandoned on account of the damage to the cloths. The washing of the press cake reduces its sucrose content to about 1 per cent. or lower. In a few factories, instead of washing the press cake, it is reduced to a thin cream with warm water and again filter-pressed.

47. After washing the cake, the presses are opened and their contents are discharged either into a car, for removal from the factory, or into a mixer, where they are reduced to a cream with water and then run into a settling pond. At the close of the season, the water is decanted from the precipitate, which soon dries and may be removed with scrapers. Certain American factories that use the Steffen lime-saccharate process calcine the press cake in Bauer's rotary kiln and recover the lime and carbonic acid for further use.

Jute or cotton cloths are used in dressing the presses. These cloths are usually changed about once a week, and the soiled cloths, after being washed, may be used again.

Special presses having very shallow recesses are often used for filtering sulphured juices, although mechanical filters with cloth pockets are usually preferred.

48. Mechanical Filters.—There are many types of the so-called mechanical filters, but they may be divided into three general classes, as follows: (1) Filters in which the liquor passes from the inside to the outside of a bag, or pocket; (2) filters in which the precipitates collect on the outside of a bag and the clear juice is drawn off from inside of it; and (3) filters in which sand or other granular material is used as a filtering medium.

49. Filters of the first class are seldom used in sugar factories. Such filters are employed in refineries, and will be described in connection with the methods of refining.

50. Filters of the second class are very extensively employed in the filtration of both juices and sirups. The Danek type of filter of this class is used by many factories in America. This filter consists of an iron box, in which are suspended a number of cotton bags, or pockets. Each bag is slipped over a metal sheet, in which are punched rectangular openings. The metal is not entirely removed from the openings, but is formed into a bow-shaped piece, which keeps the cloth away from the plate. The openings are very close to one another, and the bow-shaped guards keep the pocket distended and permit a free circulation of the liquid within the bag. The upper end of the metal sheet is attached to a longitudinally slotted pipe, one end of which is closed while the other projects beyond the bag for the delivery of the juice. The pocket is clamped to the pipe in such a way that nothing can enter it except through the pores of the cloth. On one side of the filter box are openings, into which the delivery pipes of the pockets enter and connect with discharge cocks on the outside. These cocks deliver the filtrate into a gutter. The delivery pipes have inclined planes at their ends, opposite the cocks, and these planes bear on corresponding planes in the side of the filter box, so that the weight of the filtering element itself is sufficient to press the pipe against suitable pack-

ing and thus form a tight joint. The part of the chamber below the pockets is deep and serves to collect the accumulations of mud. The filter box has a counter-poised hinged cover that closes it tightly. A pipe enters near the bottom of the pockets for the admission of cloudy juice, and a larger pipe leaves the box at the bottom for the discharge of accumulated mud. When the box is open, the filtering elements may be readily lifted from it for cleaning purposes; and they may be easily replaced owing to the inclined planes, which force them into position. A suitable device prevents the elements from coming in contact with one another or from being lifted by the current of liquor.

51. In using the filter, the liquor is admitted to the box from a tank under a head of 2 to 3 meters. The liquid passes into the pocket, on the outside of which the mud accumulates and then falls to the bottom of the box. The juice passes into the slotted pipe and is drawn off at the cocks. The mud is removed from time to time through the pipe at the bottom of the box and is filter-pressed. Low pressure is essential in the filtration with this type of filter, to prevent the mud from compacting on the cloth and rendering it impervious to liquors.

Several filters of this type are used that differ from the Danek only in the method of attaching and distending the pockets or in other mechanical details. The pockets are usually 70 cm.×70 cm. in size, and vary in number from twenty upwards.

52. Filters of the third class, using sand or other material as filtering medium have found little application in the beet-sugar industry of this country and only the Breitfeld-Danek and the Abraham sand filters as the best known in this class will be treated here.

Bone black was formerly employed in all beet-sugar factories for the filtration of the juice and sirup. This material, however, is no longer used, sulphurous acid for bleaching and mechanical filtration having displaced it.

53. General Remarks About the Filtering Apparatus.—Plate and frame presses do excellent work, but require much hand labor. The Kelly presses, which are power presses with application of Danek principles, use less hand labor, but an excess of wash water. Very recently continuous vacuum filters have been tried, but are still considered as experiments in this industry.

CONCENTRATION OF THE JUICE

54. Evaporators.—After purification the juice is ready to be concentrated. This is done in all beet-sugar factories in multiple-effect evaporators.

The amount of heat required to raise the temperature of 1 kilogram of water 1° C. is called a calorie. If 1 kilogram of water at 0° C. is heated to 100° C., 100 calories are used. By further addition of heat, the water evaporates and forms 1 kilogram of steam at 100° C. To convert 1 kilogram of water at 100° C. into 1 kilogram of steam at 100° C., 537 calories are used, which disappear as far as apparent temperature is concerned and are used up in transforming the 1 kilogram of water from the liquid form into the gaseous form. If the 1 kilogram of steam is condensed to water of 100° C., these 537 calories are again freed. These 537 calories represent the latent heat of the water vapor. On account of these physical properties, steam can be used as the best carrier of heat known. If water is boiled under vacuum the boiling point sinks to below 60° C.

The multiple-effect evaporator makes use of these physical properties of steam.

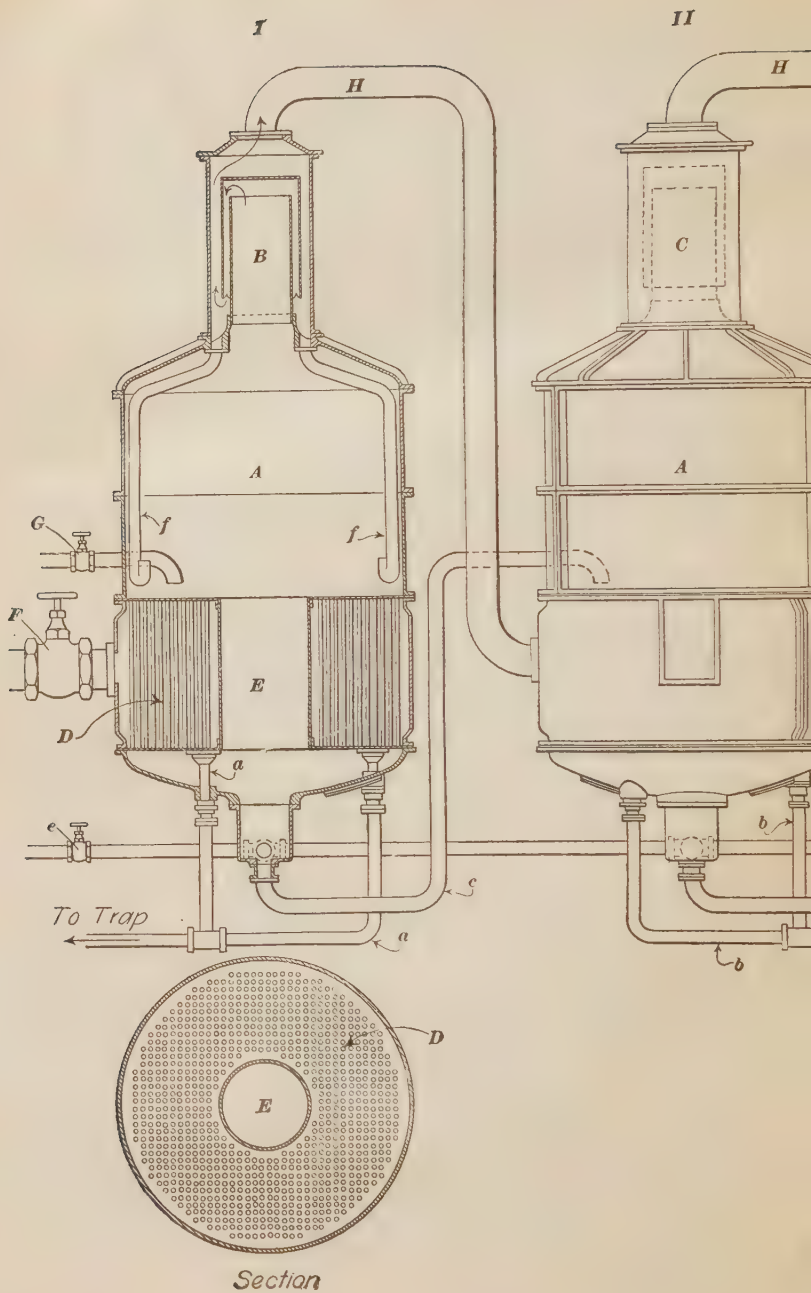
55. Multiple-Effect Evaporation.—All large factories concentrate the liquors in vacuo in **multiple effect**. With multiple-effect evaporators, as the name indicates, the heat applied in boiling the liquor is not lost after one application, but, in the form of the steam generated in this work, the heat is made to evaporate the liquid in a second vessel, and so on through several applications. In order to accomplish this

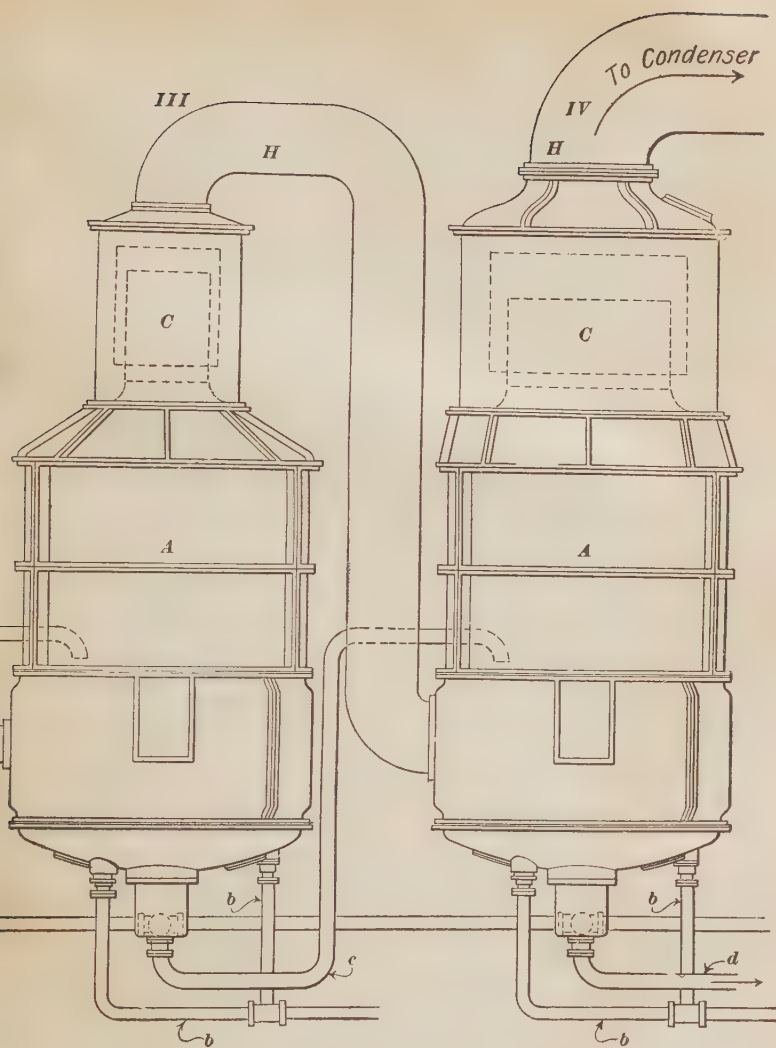
multiple application of the heat, the vessels, or pans, are closed and the air is partly exhausted from them, the vacuum increasing from pan to pan, thus reducing the temperature at which the liquid boils. The steam generated in boiling the liquid in the first vessel passes through a suitable pipe to the heating surface of the second pan, where it is condensed in evaporating the liquid in this vessel, and thus produces a partial vacuum in the first pan of the system. A similar utilization of the vapor generated in the second vessel is applied in the third, and a partial vacuum is produced in the second vessel. The last vessel of the series is connected, through a condenser, with a vacuum pump, which produces a very high vacuum in this pan. The water of condensation is led off from the first pan through a trap, and is removed from the others by pumps. This water is generally utilized in feeding the boilers, so, besides furnishing a very pure water for this purpose, it supplies the boilers with water that is partly heated.

56. It is evident that the economy of fuel with multiple-effect evaporation is very large. Roughly speaking, assuming that fuel equivalent to 1 pound of coal burned under a boiler will evaporate $7\frac{1}{2}$ pounds of water, in a multiple effect composed of two vessels, it will evaporate 15 pounds of water, and in a system of three pans, $22\frac{1}{2}$ pounds. These figures are only rough approximations. Exhaust steam from the various pumps and engines of the factory is used for multiple-effect evaporation.

57. Kinds of Multiple Effects.—Multiple effects are usually composed of four or five vessels, and are termed, respectively, *quadruple* or *quintuple effects*. At the present time, double and triple effects are seldom used. The quadruple effect is usually installed in modern factories, though many large establishments use quintuple effects. A larger number of vessels than five in a series is rarely employed.

There are many types of multiple effects in use in the sugar and other industries, but the principle on which these are based is the same for all. The form of multiple effect usually employed in the sugar industry is shown in Fig. 12, and is





called the **standard type of evaporator**. Since it is very important that the construction and manipulations of a multiple effect should be understood, this apparatus will be described in detail.

58. Quadruple-Effect Evaporator.—The evaporator shown in Fig. 12 is based on the drawings of a large quadruple effect, having 12,500 square feet of heating surface, that was designed and constructed by Samuel Vickess, M. E., New York. Only the parts of the apparatus essential to an understanding of its construction and manipulation are shown in the illustration. The dimensions are given to illustrate the variations in the construction of certain parts of the vessels that were necessary on account of the differences in vacuum.

59. Referring to Fig. 12, *A, A, A, A* are cylindrical cast-iron vessels, or pans, each 9 feet 3 inches in diameter. At *D* of each vessel there is a drum, or belt, fitted with a large number of brass tubes and a large iron tube *E*. Each brass tube is 5 feet $1\frac{1}{2}$ inches long by $1\frac{1}{2}$ inches in diameter, and the tube *E* is 3 feet in diameter. The drum *D* is called the *calandria*, and the tube *E*, the *down-take*. A section of the vessel through the calandria shows the arrangement of brass heating tubes *D*, and the down-take *E*.

60. When the apparatus is in operation, fine particles of juice are projected from the boiling liquor and are carried upwards with the vapors. This is termed *entrainment*. To avoid the loss of this juice, save-alls are located in the vapor pipes at *B, C, C, C*. The vapors follow the path in the save-all indicated by the arrows. As shown at *B*, a hood with serrations on its lower edges fits over the neck of the vessel. The particles of liquid entrained with the vapors strike against the walls of the hood and are held there until they drip to its lower edge. Here, the serrations gather them into large drops, which fall into a suitable canal and are led back into the vessel through the pipes *f*. These pipes are curved at the lower ends, so as to trap a portion of the liquid and thus prevent the vapors from passing through them.

The vessels are connected with one another by pipes *H*, which lead from the vapor space of one to the calandria of the next, with the exception of the vapor pipe of the last pan, which is connected, through a condenser, with a powerful vacuum pump. The types of vacuum pumps will be described later.

61. Since, as just explained, the first vessel of a quadruple effect is usually operated under low pressure and the others with a vacuum, which increases from pan to pan, the volumes of the vapors from equal quantities of boiling liquor also increase from pan to pan, being greater the higher the vacuum. Provision for this increase in volume is made by increasing the size of the save-alls *C* and the vapor pipes *H*. The diameters of the necks of the vessels at the save-alls *B*, *C* in the apparatus, Fig. 12, are, respectively, 2 feet, 2 feet 2 inches, 2 feet 9 inches, and 5 feet 6 inches, and the corresponding dimensions of the vapor pipes *H* of the first three effects are 18 inches, 20 inches, and 46 inches. If it were not for this enlargement of these parts of the apparatus, the vapors would have to travel at a very high velocity, the entrainment of juice would be excessive, and the vapors could not escape with sufficient rapidity.

62. Aside from the loss of sugar involved, entrainment is very objectionable, since the water obtained from the condensation of the vapors is used in feeding the steam boilers. When the feedwater contains an appreciable quantity of sugar, foaming is produced, which endangers the safety of the boiler.

The upper part of the steam space of each calandria is connected by means of a small pipe, which is provided with suitable regulating valves, either with the vapor pipe of the last effect or with its own vapor pipe. The purpose of these pipes is to remove the ammoniacal gas that results from the decomposition of certain nitrogenous constituents of the juice. The gas is lighter than the steam; hence it rises to the upper part of the calandria. The quantity of the gas in beet-sugar manufacture is so large that where proper provisions for its removal are not made, the tubes of the evaporator may be destroyed.

63. The remaining details of the construction of the quadruple effect will be explained in the following description of the manipulation of the apparatus:

Referring to Fig. 12, the travel of the juice and vapors follows the order of the Roman numerals. To begin work with the apparatus empty, the vacuum pump is started and juice is admitted into vessel *I* through pipe *G*, into *II* through pipe *c*, and so on. The pipes *G* and *c* are provided with valves so as to regulate the flow of the juice. Having filled each pan with juice to a point a little below the tops of tubes, steam is turned into the calandria of the first pan through the valve *F*. The steam used for this purpose is that exhausted by the various engines of the factory. Sufficient pressure is secured by the use of a back-pressure valve, and deficiencies in the supply of exhaust steam are made up with live steam. The juice in the first pan soon begins to boil, and the vapor generated passes through the vapor pipe and into the calandria of the second pan, where it is condensed in boiling the liquor in this vessel; in a like manner, the juice is boiled in the third and fourth pans. The vacuum in the last pan is produced by the condensation of the vapors from that vessel and the assistance of the vacuum pump; that in the second and third pans, by the condensation of their vapors in the calandrias of the third and fourth pans, respectively; and in the first, or pressure, pan, as it is often termed, the pressure or vacuum maintained is regulated by the amount of steam admitted at the valve *F*.

The condensation water from the calandria of vessel *I* flows through the pipes *a* and a steam trap to the feedwater tank of the boilers; that from the remaining pans is removed by means of pumps.

64. The evaporation having been started as described, the level of the boiling liquor in each vessel should be maintained just high enough to cover the tubes of the calandria. This is done by adjusting the flow of juice through the pipes *G* and *c* by means of the valves at each pan. The thick juice is pumped from the last pan through the pipe *d* into

4th EFFECT

3^d EFFECT

2^d EFFECT

1st EFFECT

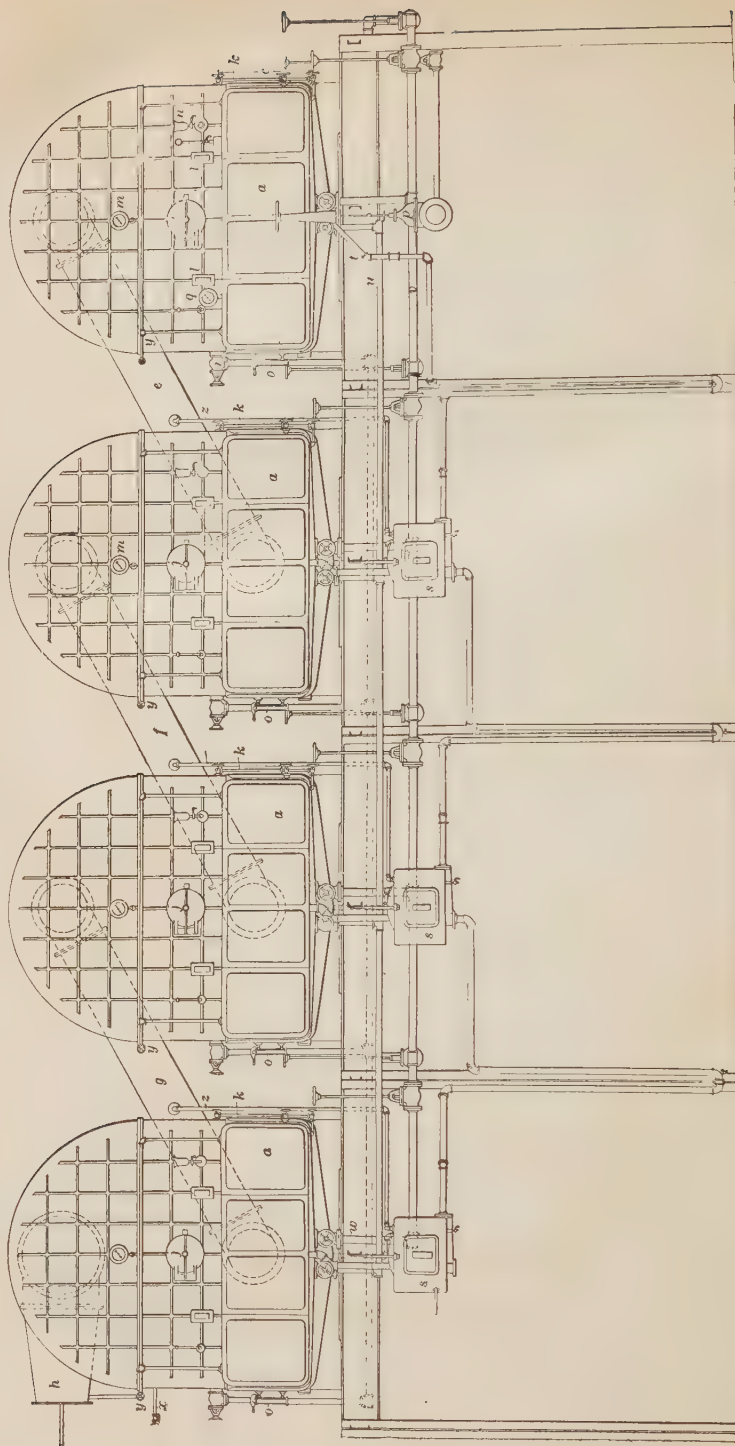


FIG. 13

the storage tanks. The flow of the thick juice to the pump is regulated by a valve in pipe *d*. This valve is opened or closed a little at a time until the thick juice of the desired density is obtained. It should be noted that a current of thin juice flows through the apparatus from pan to pan, the thin, fresh juice entering at the pipe *G* and the thick juice being pumped out of

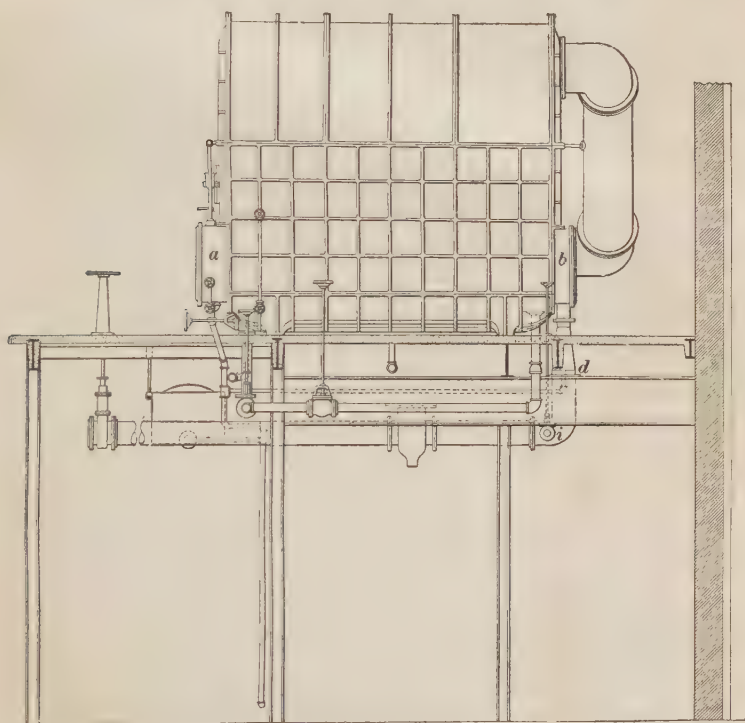


FIG. 14

the last vessel at *d*. The process is therefore a continuous one.

The steam pressure in the calandria of vessel *I* is kept as low as is consistent with obtaining sufficient service from the apparatus and maintaining a regular flow of liquor from pan to pan. Under ordinary working conditions, the gauges connected with the vapor space of the vessels will show the following indications: *I*, 2 to 3 inches (mercury) pressure;

II, 5 to 7 inches vacuum; *III*, 15 to 16 inches vacuum; *IV*, 27+inches vacuum. The vacuum in the fourth pan should be as high as the condenser and pump will produce. The pressure in the first pan and the vacuums in the second and third are regulated by admission of steam to the calandria of the first pan.

65. A modified **Welner-Jelinek quadruple effect**, manufactured by E. H. Dyer and Company, is shown in Figs. 13 and 14. The principle of the multiple effect and the method of operating it have been previously described. Therefore, only a general description of a modified Welner-Jelinek apparatus will be given. The effects, or pans, are rectangular cast-iron vessels. The heating surface is composed of horizontal brass or copper tubes of small diameter that enter the heads, or distributing steam chambers, *a* and *b*. In this type of multiple effect, the steam circulates through the tubes, whereas in the standard evaporator, the juice fills the tubes and the steam surrounds them. The scale produced by the precipitation of certain impurities in the juice forms on the outside of the tubes. This scale can be removed either by spraying cold water on the tubes after heating them or by boiling caustic soda in the pan and following this with dilute hydrochloric acid. Scraping is also resorted to in cleaning the tubes.

In the evaporation of beet juices, a considerable evolution of ammonia arises from the decomposition of the nitrogenous constituents. The pipes *y* leading from the steam chambers to the vapor pipes are for the purpose of removing this ammonia. The ammonia rapidly destroys the copper tubes, especially those in the upper rows. In the standard type of evaporator, the ammonia attacks the upper ends of the tubes.

66. Explanation of Evaporator.—In the following list, the parts of the apparatus indicated by letters in Figs. 13 and 14 are given:

a, b, chambers for the distribution of the steam and the collection of the water of condensation.

c, line indicating the depth of the juice space.

d, main for bringing exhaust steam from the engines and pumps to the heating surface of the first effect.

e, f, g, vapor pipes of the first, second, and third effects.

h, vapor pipe of the fourth effect. This pipe leads to the condenser.

i, live-steam pipe, for use when cleaning the heating surfaces or when starting the apparatus for heating the liquors.

j, manholes, to give access to the effects for cleaning or for repairs.

k, gauge glasses for juice. The small gauge glass below *a*, Fig. 14, is to note the quantity of the condensation water remaining in the vessel.

l, eyeglasses, for watching the evaporation.

m, vacuum gauges.

n, butter cup, for injecting oil or grease so as to reduce frothing.

o, cups for drawing and examining samples of the liquors. The test samples are usually drawn only from the last effect.

p, main steam valve.

q, steam gauge.

r, water valve.

s, steam traps.

t, pipe-connecting pans and traps.

u, wash-out pipe.

v, pipe to convey liquor from pan to pan. This pipe is used in connection with charge pipes, not shown in the figures.

w, pipe leading from the fourth effect to the sirup pump, for the removal of finished liquor. This pipe is also connected with all the effects, for use in emptying them.

x, pipe connected with the sirup pump, for equalizing the vacuum in the last effect and the pump.

y, pipes for the removal of the ammoniacal vapors from the steam chambers.

z, pipes connecting the traps and vapor pipes.

CLEANING THE EVAPORATORS

67. After being in use for several weeks, the evaporators become incrustated with scale, which consists of carbonate, sulphate, silicate, oxalate of lime, and other impurities.

Part of these impurities are soluble in hydrochloric acid and part have to be converted into carbonates before boiling with acid.

In practice, a strong soda-ash solution is boiled in the pans for two or three hours. The soda solution is then run into a storage tank for future use and the heating surfaces are rinsed with water. The action of the soda softens the scales, but dissolves very little of this material. This soda treatment has to be followed by boiling with dilute hydrochloric acid. Both the soda and the acid should be boiled with the pans open, that is, at atmospheric pressure. The spent acid is run into the sewer. The soda solution may be used many times by decanting it from the mud, which settles to the bottom of the tank, and occasionally adding fresh caustic soda to it.

68. The two described systems of evaporators, the standard vertical and the Welner-Jelinek evaporators are quadruple effects. In recent years quintuple effects are almost generally adopted in the beet-sugar industry. The principle, of course, remains the same, only the steam economy is increased in a quintuple effect.

This steam economy can be further essentially increased by using vapors from the first and second body for heating the diffusion battery and the diffusion juice after the battery and the thin juices between the different filtration stations.

69. The steam economy in a quintuple effect heating the battery and the diffusion juice with vapors from the second body and the thin juice with vapor from the first body over a quadruple effect without using vapor for heating purposes, is about 17 to 20 per cent. figured on the total steam and consequently, coal consumption of the house. In other words, if a house with a quadruple effect evaporator and no vapor heaters would use 14 per cent. of a certain coal, it would use

11.2 per cent. of the same coal after changing to a quintuple effect evaporating system and vapor heaters.

70. Purification of the Thick Juices.—As it comes from the multiple effect, the thick juice contains many impurities in suspension that must be removed. This is accomplished by settling and decantation or, preferably, by filtration. Mechanical filters that use either cloth or sand are employed for this filtration, or plate-and-frame presses.

Prior to filtration, the thick juice is sulphured for bleaching purposes. A very small quantity of lime may be added to the thick juice before sulphuring. The juice is then heated by *blowing in* of steam. The tanks in which this heating and sulphuring is done are called the *blow-ups*. The alkalinity of the sulphured thick juice should be .010 to .005.

In white-sugar factories—and all American factories are white-sugar factories—the thick juice filtration is the last filtration before the boiling to grain in the vacuum pan, and therefore should be perfect to produce perfectly white sugar.

MANUFACTURE OF BEET SUGAR

(PART 2)

CRYSTALLIZATION

1. The thick juice obtained by the evaporation of the juice, as it comes from the thick-juice presses, usually has a density of about 60° Brix at 17½° C. and contains about 40 per cent. of water.

In all modern factories, the sugar is *crystallized*, or *grained*, in a single-effect, vacuum evaporator, termed a *vacuum pan*, and this crystallization is effected while the liquor is boiling. The vacuum-pan process of crystallizing sugar has resulted in great economy in the manufacture and in the direct production of high-grade sugars.

The apparatus used and the methods of procedure followed in boiling vacuum-pan sugars will now be described.

2. **Vacuum Pan.**—A vacuum pan, shown in Fig. 1, consists of a cylindrical cast-iron or copper vessel with a conical bottom and dome-shaped top. Copper, however, is rarely used in this construction. A vapor pipe *A* leads from the dome to a save-all *B*, and from this point to a vacuum pump. A large valve, called the *strike*, or *foot-valve*, is located at the bottom of the pan at *C*. This valve is used for discharging the *massecuite*, which consists of a mass of sugar crystals and molasses. A series of copper coils—usually four or more in number—fitted with valves *D* forms the heating surface. A large opening, or well, similar to the down-take of the multiple effect, is provided at the centers of the coils.

This well serves to promote the circulation of the massecuite and to give access to the pan for repairs. The coils are drained of condensation water through tail-pipes and steam traps.

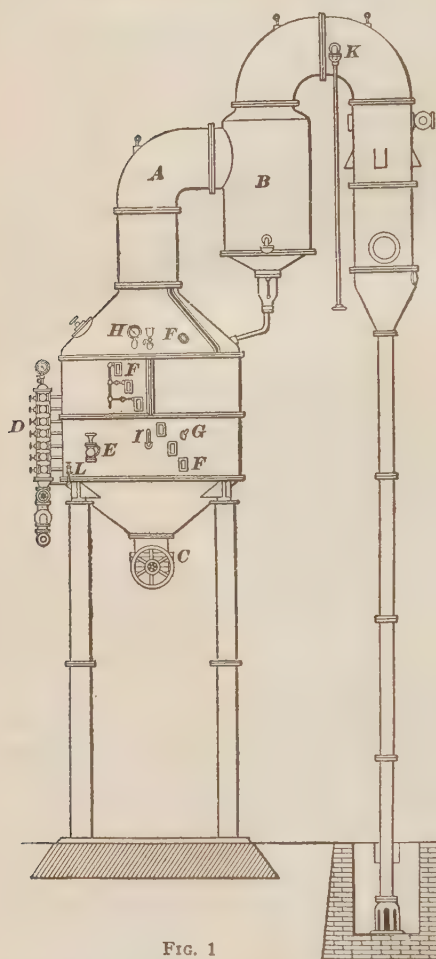


FIG. 1

A liquor charge pipe *E* enters the pan either near the strike valve or at a higher point, and in the latter case, passes downwards almost to the bottom of the vessel. This pipe has branches that connect with the pan storage tanks. Eyeglasses *F* for watching the progress of the work, a proof stick *G* for withdrawing samples of the material examination, a vacuum gauge *H*, a thermometer *I*, a break-vacuum valve *K*, and a steaming-out pipe *L* complete the usual equipment of the pan.

3. Many factories have a central condenser with which all the pans and multiple effects are connected. With this type of installation, a head-valve is placed in the

vapor pipe, to be closed when the pan is not in use. A small pipe connects the pan with the vacuum line, for the purpose of establishing the same pressure on both sides of the head-

valve when a *strike* of sugar is to be boiled. Pans are also occasionally arranged for the discharge of the massecuite by air pressure and its distribution among the crystallizers through large pipes. This arrangement also requires a head-valve in the vapor pipe.

4. There are two types of pumps employed to produce the vacuum, namely, that of the wet system and that of the dry system. In the wet system, the water of condensation from the vapors and the condensing water are passed through the pump. In the dry system, the condensation and condensing waters are led off through a Torricellian tube, or *leg pipe*, the lower end of which dips below the surface of water into a small tank, called the *hot-well*. The water in this well seals the pipe and at the same time permits the escape of water from the condenser. A small pipe connects the condenser with the vacuum pump, which removes the air from the pan when beginning an operation, or *strike*, and also the air that enters the apparatus by leakage or that which is entrained by the sirup.

5. The vacuum in the pan is increased or decreased by increasing or decreasing the quantity of water injected into the condenser. This regulation of the vacuum is of importance, since the temperature of the massecuite must be varied at different stages of the operation of boiling the sugar.

The heating surfaces of the vacuum pans also get dirty or scale, but not so rapidly as those of the evaporators. These surfaces may be readily cleaned by boiling a strong caustic soda and carbonate solution in the pan and then following with hydrochloric acid.

TERMS USED IN SUGAR BOILING

6. The juice coming from the evaporators is the *evaporator thick juice* (purity 88-90). This juice is treated in the *blow-ups*, is filtered and goes to the pan storage tanks as *pan storage thick juice*. In modern beet sugar factories the extraction of sugar by crystallizing is done in two boiling operations. From

the *first massecuite*, or *first fillmass*, or *white fillmass*, (purity 85–87), the *granulated sugar* is obtained. The sirup spun from the granulated sugar is called *green sirup*, or *high green sirup* (purity 74–76). After this sirup is spun off, the sugar is washed and the resulting sirup is the *high-wash sirup* (purity 85–88). This high-wash sirup is boiled back into the first fillmass. The green sirup is boiled to the *second fillmass*, or *remelt fillmass*, or *brown fillmass* (purity around 75), which is *grained* or *boiled to grain* and sent to the crystallizers. From there it is emptied into a *brown sugar mixer* and separated in centrifugal machines into *brown sugar*, or *remelt sugar*, which is washed, dissolved in thin juice, and boiled together with the thick juice into the first fillmass. The sirup coming from the brown sugar is called *low green sirup*, or *molasses* (purity around 60), a sirup from which no more sugar can be extracted by simple concentration; and *low wash sirup*, which is boiled back again into the brown fillmass.

7. When thin sugar solutions are concentrated they become *saturated* with sugar. This saturation point is the point where a solution does not dissolve any more sugar and no sugar is separated out by crystallization. The saturation point rises with the temperature. A saturated sugar solution at 20° C. holds about 66 per cent. sugar in solution.

If a saturated sugar solution is further concentrated, it does not immediately separate sugar crystals (the process of crystallization taking time), but it becomes *supersaturated* until the supersaturation reaches a certain limit, when crystals start to form. If left alone in this state, enough sugar crystals are formed in a short time to leave the remaining solution in a saturated state. The ratio of the amount of sugar in a supersaturated solution to that in a saturated solution, the water content and temperature being identical in both cases, is known as the *supersaturation coefficient*. To keep the right degree of supersaturation during the boiling process, is the secret of sugar boiling.

METHODS OF SUGAR BOILING

8. Vacuum-Pan Process.—Having described the apparatus in the boiling of sugar—the vacuum pan—the methods will next be considered. While it is important that the process of sugar boiling and the principles on which it is based be thoroughly understood, nevertheless, to become proficient in this work, actual practice at the vacuum pan is essential. Practice is necessary in order to learn to judge the relative densities of the liquor under different conditions of purity, vacuum, and temperature, from the appearance of the material as it is boiling in the pan and from the examination of test samples drawn with the proof-stick. Practice is also necessary in order to decide on the necessary manipulations from the quantity and appearance of the crystals in the test samples. There is a decided difference between boiling beet-sugar juices and cane-sugar juices and the main and fundamental reason for this difference is the different viscosity of beet-sugar and cane-sugar juices. Beet-sugar massecuites of the same water content as cane massecuites will move sluggishly and slowly in the pan while cane-sugar massecuites still are quite liquid at the same temperature. It is, therefore, necessary to keep the beet-sugar massecuites at a higher temperature than the cane-sugar massecuites to insure a good circulation in the pan and facilitate the deposition of sugar on the surface of the already formed crystals by this circulation, and in graining beet-sugar juices a high temperature, say 75° C., or higher, is necessary to form sharp individual crystals.

Cane-sugar boilers, accustomed to graining at between 60° to 65° and keeping their massecuite cooler than beet-sugar massecuite during the following boiling, when starting in beet-sugar factories usually turn out a dull white flowery sugar, which, besides having no sparkle, does not wash very well either. It generally takes some time for the cane-sugar boiler to recognize this characteristic of the beet juices.

9. Grained Strikes.—Having closed the foot- and break-vacuum valves of the pan, the vacuum pump is started and the circulating water is turned into the condenser. When the gauge indicates a vacuum in the pan ranging from 15 to 20 inches, the sugar boiler opens the charge valve and draws thick juice into the vessel until the copper coils are covered, or until, from previous experience, he judges that the pan contains sufficient liquid. He now carefully and slowly opens the steam valves, one at a time, beginning with the lowest, or No. 1, as it is called, allowing time for the water of condensation to escape from the coil. Care is necessary, so as to avoid injuring a coil. The thick juice soon boils and the water evaporates rapidly from it. When the surface of the liquor falls almost to the top coil, the steam valve on that coil must be closed, and so on from coil to coil, never leaving one uncovered while its steam valve is open.

10. Soon, from the appearance of the juice and from the flow of the drops projected on the eye glasses, the sugar boiler notes that the liquor is becoming very dense. By gradually closing the circulating-water valve, he raises the temperature of the boiling liquid to about 75° C., or to such other temperature as the grade of sugar which he is boiling requires, and by manipulating the water valve maintains this condition. From time to time, a test sample is drawn with the proof stick and is examined on a piece of plain glass by means of transmitted light. In a short time, the boiling liquid becomes saturated and then supersaturated with sugar, at the pan temperature, and microscopic crystals of sugar begin to form.

It requires experience on the part of the pan boiler to detect promptly these minute crystals, which an inexperienced eye would scarcely note. The sugar boiler now carefully follows the formation of the crystals, and the moment he judges that these are numerically sufficient, he opens the charge valve and draws a small quantity of thick juice into the pan; he also slightly closes the circulating-water valve so as to prevent a fall in the temperature of the boiling liquor.

A large charge of juice would cool the boiling mass and thus cause additional crystals to form, and the same thing would occur if the vacuum should rise and thus cool the liquor. The reason for this is that when a solution is too far supersaturated, additional undesired crystals separate.

11. As the charge of liquor evaporates, the panman carefully watches the liquid until it has again reached the right degree of supersaturation. By this procedure, he causes a part of the sugar in the juice to be deposited on the crystals already present, thus building up the crystals to commercial size. The formation of new, small crystals at this point results in the condition known as *false grain*. The sugar boiler frequently examines test samples on the piece of glass, for false grain, which he must immediately get rid of. This is accomplished by increasing the temperature of the pan and charging copiously with juice. The moment the disappearance of the false grain is noted, the panman cools the massecuite gradually, and when the temperature falls to about 75° C., he continues the boiling as before. False grain is objectionable, because it impedes centrifuging of the sugar. A loss of sugar also occurs when the fine crystals pass through the centrifugal sieves.

12. The alternate charging and evaporating are continued until the pan is filled with massecuite. The charges of juice are continued until the pan is about three-fourths or five-sixths full, when high green sirup is drawn in. During the latter stage of the operation, the massecuite is gradually increased in density, especially before the addition of high green sirup, and finally it is evaporated to a water content of about 7 per cent. During this process, the crystals increase from a minute size to that of commercial sugar.

The steam is now shut off from the coils, the vacuum pump is stopped, and the break-vacuum valve is opened. When the vacuum falls to zero, as indicated by the gauge, the massecuite is discharged from the pan through the foot-valve. The further treatment of the massecuite is described in the paragraphs devoted to crystallization in motion and to curing the sugar.

After boiling and discharging a strike, the pan must be thoroughly cleaned by steaming, to recover the last adhering portions of massecuite. This steaming is necessary not only to recover the sugar, but also to prevent it from caramelizing on the coils.

13. At the beginning of the strike, it is important that the pan boiler form a sufficient number of crystals and that he at all times avoids excessively large charges of juice, otherwise false grain may be formed.

The grade of sugar required and other considerations determine the point in the pan at which the strike is grained. When the crystals are formed between the first and second coils, the strike is said to be *grained low*, and when formed near the middle of the pan, to be *grained high*. Low grain-ing produces comparatively few crystals of large size, and requires long boiling. High graining, on the contrary, produces a fine-grained sugar, with short boiling.

The temperature conditions vary in boiling different grades of sugar. A soft, low-test sugar is produced by boiling the strike at as low a temperature as possible, and clean, hard crystals are obtained by hot boiling. Large crystals are more readily obtained with thinner juices and the consequent slow evaporation. The commercial granulated sugar is boiled at a high temperature when produced in the factory, and usually also when made in the refineries.

14. In the way of boiling sugar above described, that is, concentrating the original charge of thick juice until the grain appears by itself, the supersaturation of the liquor is extremely high, maybe as high as 1.5 to 1.6 and varying with the time of concentrating the charge. In connection with this high supersaturation, the liquor has a high viscosity.

Many sugar boilers do not wait until the grain comes by itself, but start the grain, or force the grain before the supersaturation reaches such a high degree, by giving the liquor a heavy mechanical movement, which may be done by taking in a very small short charge of liquor when the supersaturation coefficient is around 1.3. One short charge generally does not

produce enough grain and the operation has to be repeated several times. The sugar boiler soon finds out how often and how long he has to charge to make enough grain in his fillmass. After graining, the supersaturation coefficient is kept between 1.1 and 1.2. This method produces a sharper grain, less twin crystals and conglomerates of crystals, which can be washed easier in the centrifugals, due to a smaller degree of concentration and viscosity of the original charge.

15. Boiling Brown, or Second, or Remelt Fillmass.—

The boiling of the second fillmass from high green sirup and low wash sirup is very similar to the boiling of the first fillmass. The difference is that all liquors are of a much lower purity and therefore require more time and much more careful watching of the fillmass. The vacuum pans are either constructed like those first described with heating coils, or they are calandria pans with heating chambers similar to the evaporator heating chambers with wider circulation pipes suspended inside the pan and heated with vapor from the evaporators. This results in further steam economy. High temperatures are necessary to break the viscosity of the green sirup, which has a purity of around 76 to 78, but since the sugar crystallizes out of these liquors slowly on account of their impurities much time has to be given to the crystallization of the sugar and a comparatively small amount of steam can be applied to the pan or steam of low pressure like exhaust steam or vapor from the first effect of the evaporators is sufficient to furnish the heat for this boiling process.

From the pan the fillmass is dropped into crystallizers. These are cylinders with a double mantle for water heating or cooling and a stirrer with arms arranged in a spiral so as to keep the fillmass in motion. The time the fillmass is left in these crystallizers is from 3 to 4 days.

16. Crystallization in Motion.—

The process known as **crystallization in motion** originated with Wulf, in Germany, in 1884, and the first practical apparatus for use was devised by Stammer and Bock. Crystallization in motion is now used in all beet-sugar factories. The process consists

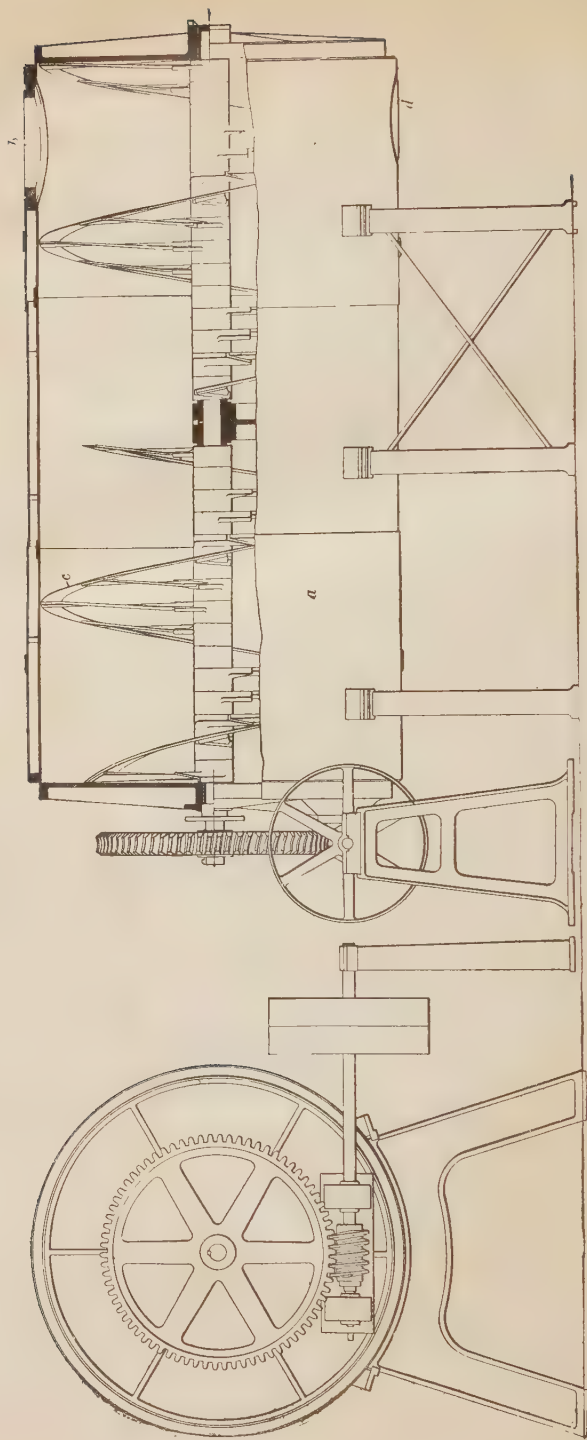


FIG. 2

essentially in forming the crystals of sugar in a vacuum pan and then emptying the fillmass into a crystallizer and keeping it in motion until the crystallization is completed. It is always preferable to carry the crystallization as far as possible in the vacuum pan and then continue it in the crystallizer.

17. Many modifications of the original crystallizer are in use. A common form of crystallizer is shown in Fig. 2. This device consists of a horizontal iron cylinder *a* provided with a spiral mixer *c*, which is driven by means of gearing, as shown. At *b* is shown an opening for charging the apparatus and at *d*, a gate, or valve, for discharging it. The massecuite is sometimes discharged through a pipe leading to the centrifugals by means of compressed air. The apparatus is usually fitted with a water-jacket, which assists in regulating the temperature of the massecuite. The arrangement of the spiral mixer is such that all parts of the massecuite can be kept in motion.

In charging, the apparatus should be as warm as the massecuite, and the temperature and density of the latter should be so regulated that new crystals, or *false grain*, will not form.

Occasionally, open crystallizers are used, but these are not usually considered to be so effective as the type described, because the temperature cannot be so readily regulated.

18. Another type of apparatus, the **Grosse vacuum crystallizer**, is shown in Fig. 3. This device resembles a vacuum pan in general construction, but differs from it in having a screw in the tube *a* driven by the gearing *b*. This screw conveys the massecuite from the lower part of the crystallizer to the upper part of the apparatus, where it falls on an umbrella-shaped deflector and is diverted back into the body of the massecuite. The object of this is to bring fresh portions of the sugar solution into contact with the crystals and to promote the development of the latter. The vapor pipe *c* connects the crystallizer with the vacuum pump through a condenser. It is claimed that crystallization is accomplished more quickly in a vacuum-pan crystallizer of this type than in the ordinary vacuum pan supplemented by crystallization in motion.

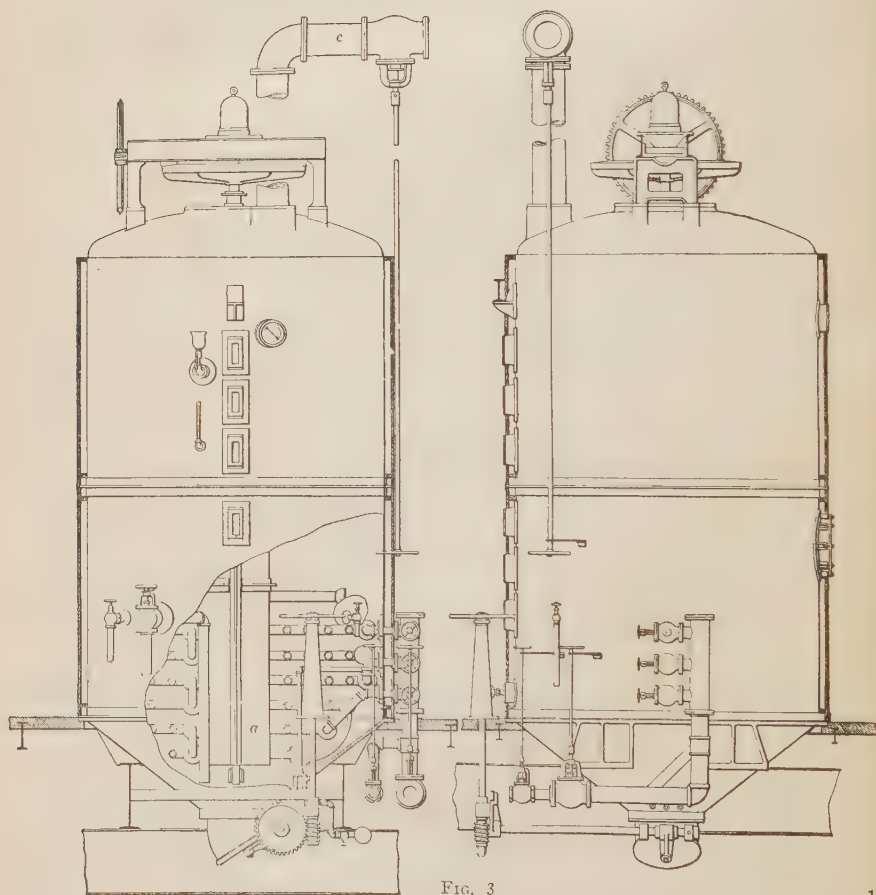
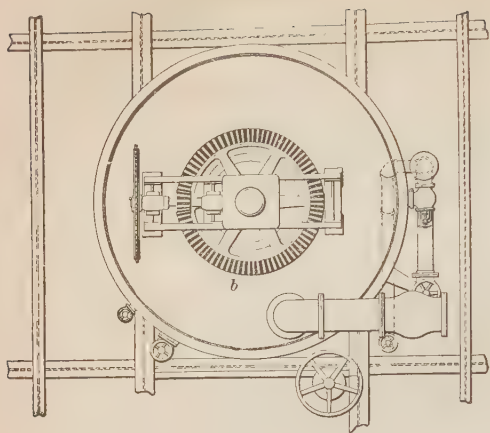


FIG. 3

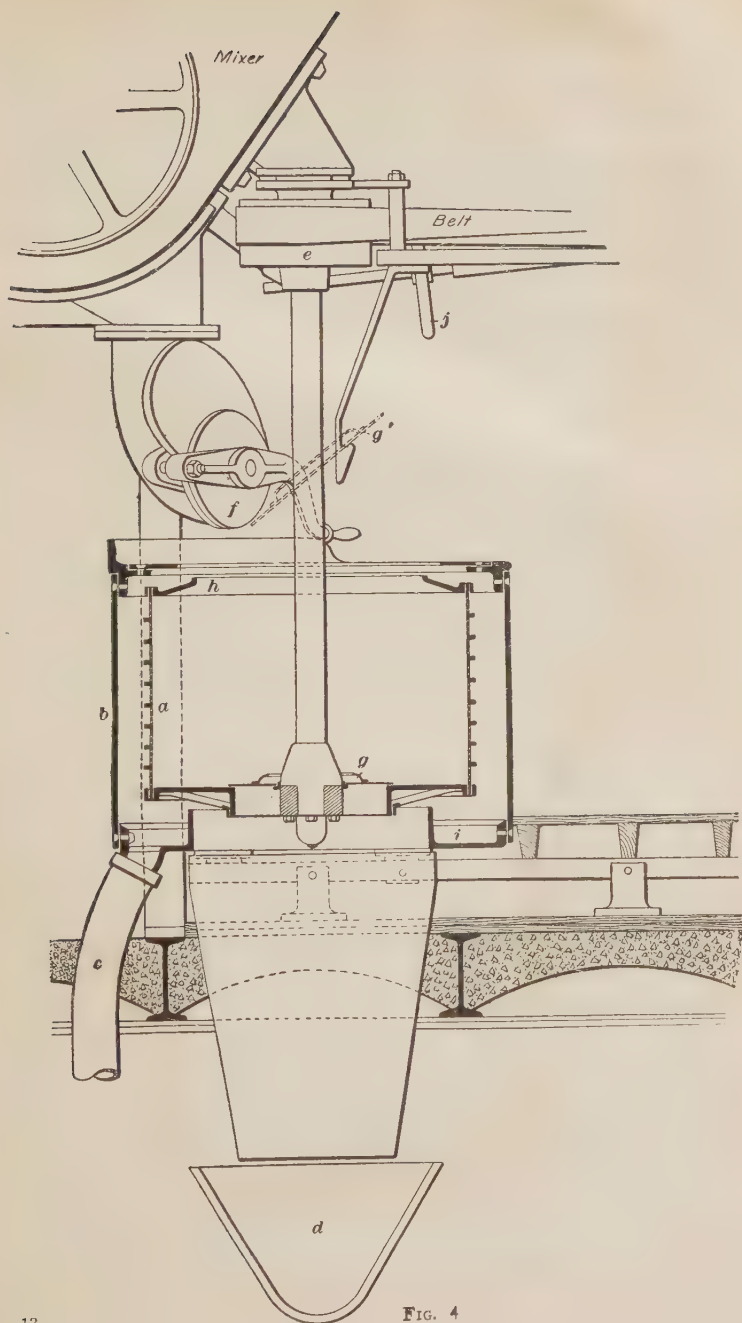


FIG. 4

CURING THE SUGAR

19. In curing the sugar, the massecuite is discharged directly from the vacuum pan or from a crystallizer into the mixer. It is then fed into the centrifugals from the mixer as required for purging.

A sectional view of a centrifugal and its accessories is shown in Fig. 4. The machine illustrated is of the Weston type. This centrifugal consists of a round, perforated metal basket *a* suspended by a spindle, or shaft, from a lug attached to the mixer. The usual diameters of centrifugal baskets are 30, 36, and 40 inches. At the upper end of the spindle is a bearing and pulley *e*, which is driven by a belt from a countershaft. The massecuite in the basket is prevented from overflowing by the rim *h*, which limits the size of the charge. The basket is lined with a very fine woven-wire or sheet-metal sieve. Brass or copper is used in making the lining. The basket is encircled by a stationary casing, or monitor, *b*. Centrifugals are frequently driven by water or electric motors attached directly to the head of the spindle.

20. In operating the centrifugal, the gate *f* is opened and a charge of massecuite is run into the basket, which is revolved slowly by hand or more rapidly by power, to distribute the charge. After charging, the friction clutch (not shown) that connects the machine with the driving pulley is thrown into position and the centrifugal is revolved at a rate of speed depending on the diameter of the basket. The usual speed for a 30-inch basket is from 1,200 to 1,400 revolutions per minute. The centrifugal force throws the massecuite against the perforated wall of the basket *a* and carries it upwards to the rim *h*; the sirups or molasses leaves the crystals, passes through the perforations, runs down the inside of the casing into the gutter *i*, and then passes through the pipe *c* to the storage tank. If only raw sugar is being made, the operator, when he judges from the elapsed time and the appearance of the wall of sugar that the latter is dry, that is, practically free of sirups or molasses, releases the friction clutch and applies the brake *j*

to stop the machine. If white sugar is being made, the operator, when he notes that the charge is practically free from green sirup, sprays water on the wall of sugar from a sprinkling can, syringe, or other device while the machine is revolving. When the charge of sugar is dry, the machine is stopped and the *butterfly*, or valve, *g* is lifted to the position shown by the dotted lines at *g'*. The sugar is then *cut down* and discharged through the bottom of the machine into the conveyer *d*.

Centrifugals are often provided with a spout *c* that can be moved from one trough to another, in order to separate the green sirup from the wash sirup. This arrangement is not entirely satisfactory, as the wash sirup carries with it some green sirup adhering to the casing *b* and the gutter *i*. This difficulty, however, is overcome in some machines by patented devices.

When discharged from the centrifugal, the sugar, as just mentioned, falls into a conveyer. It is then elevated to the wet sugar storage bins. From these bins it passes through a dryer, or granulator, from which it emerges as commercial granulated sugar.

21. Granulator.—The *granulator* (see *t*, Fig. 5) is an inclined, revolving iron cylinder about 25 feet in length and about 5 feet in diameter. Shallow shelves are attached longitudinally to the inside of the cylinder, and the apparatus is so fitted that a current of hot air may be drawn through it by an exhaust fan. The sugar is conveyed into the cylinder at the elevated end and is carried part way through a revolution of the apparatus by the shelves, from which it then falls in a shower. The current of hot air from the lower end of the cylinder passes through the shower of sugar and dries it. Owing to the inclined position of the cylinder, the sugar travels through it to its lower end and there falls into revolving sieves, which remove the lumps and divide the sugar into the commercial sizes of granulated sugar. The motion imparted to the sugar in passing through the granulator separates the crystals from one another; hence, the name of the machine.

PROCESSES FOR EXTRACTING SUGAR FROM THE MOLASSES

22. The Molasses.—The molasses of the beet, in which the sugar cannot be crystallized, contains from one and a half to two times as much sugar as the corresponding molasses of the sugar cane. This large sugar content is due to the quantity and nature of the organic and mineral impurities in solution in the molasses. In order to recover part or practically all of the sugar remaining in the molasses, either a portion of the impurities must be removed or the sugar itself must be precipitated. The former is accomplished by the *osmose process*, which is based on the same properties of the sugar and those of the impurities as the diffusion process used in extracting the juice from the beet. In the *precipitation process*, lime and strontia are the usual precipitants.

The composition of a sample of beet molasses is shown in the following analysis:

Sucrose	50%
Raffinose	Trace
Organic matter (not sugar)	22%
Ash (mineral matter)	11%
Water	17%
<hr/>	
Total	100%

OSMOSE PROCESS

23. The *osmose process* is an application of dialysis to beet molasses. Artificial parchment, which supplies the membrane, is held in position between the frames of an apparatus that somewhat resembles a filter press. Water is circulated in compartments formed by the parchment, on each side of which a dilute solution of the molasses also circulates. As previously described, certain bodies called *crystalloids* readily pass through a membrane, from one solu-

tion into a contiguous solution, while other bodies, known as *colloids*, do not pass the membrane at all. The crystalloids do not pass through the membrane with equal facility, certain salts passing with much greater rapidity than sucrose. If the liquids on both sides of the membrane are left in contact long enough, an equilibrium will be established as regards the crystalloids, which are present in like proportion in the two solutions. Of the crystalloids in the molasses, the salts pass the parchment membrane faster than the sugar. As it is desirable to retain the sucrose in the diluted molasses and to pass the maximum practicable proportion of the salts through the membrane, the liquids are circulated somewhat rapidly and are heated to the temperature favorable to the dialysis.

24. In practice, the water abstracts a part of the sucrose and about two-thirds of the saline matter. The dilute molasses from the *osmogene*, as the apparatus is often termed, is concentrated in the multiple effect and is again boiled in the vacuum pan. A part of the sugar is thus recovered by crystallization. This crystallization is made possible by the removal of the highly melassigenic salts in the osmose water that prevent the formation of crystals. It is estimated that the salts present in the molasses prevent about three and one-half times their weight of sucrose from crystallizing.

The Vallez triple osmose process is said to be especially effective and is used extensively in the American factories.

Prior to osmosis, any lime that is present should be precipitated with carbonate of soda or with acid phosphate of barium. If the solution is acid, neutral, or only slightly alkaline, caustic soda should be added to it.

At present the osmose process is comparatively little used and belongs to history.

PRECIPITATION, OR SACCHARATE, PROCESSES

25. The precipitation, or saccharate, processes are the opposite of the osmose process, as they depend on the precipitation of the sucrose as a saccharate, thus removing it from the solution, instead of on the removal of the impurities. Several precipitation processes have been devised, but those usually applied depend on lime or strontia as the precipitant.

Calcium, Ca, forms three saccharates, or sucrates, with sucrose, of which the monobasic and tribasic are technically the most important:

Monobasic saccharate, $(C_{12}H_{22}O_{11}) \cdot CaO$

Dibasic saccharate, $(C_{12}H_{22}O_{11}) \cdot 2CaO$

Tribasic saccharate, $(C_{12}H_{22}O_{11}) \cdot 3CaO$

The monobasic saccharate is soluble in water and insoluble in alcohol. On heating the water solution to boiling, all the lime combines with one-third of the sucrose to form the tribasic saccharate, which is not readily soluble in water. The dibasic saccharate is soluble in warm water, but in cold water, it separates in the crystalline form.

26. If 3 molecules of lime is added to the sugar solution, temperature being kept below 30° C., the tribasic saccharate will be precipitated. As will be noted later in the description of the Steffen separation process, the temperature of the solution has a marked influence on the physical condition of the precipitate. This saccharate is soluble with difficulty in 200 parts of water and is insoluble in a solution of calcium hydrate.

27. *Strontium, barium*, and *lead* also form saccharates with sucrose, but not so many as the lime.

Strontium forms two saccharates—the monobasic and the dibasic. The monobasic is formed in the cold, while the

dibasic is formed at 100° C. or more readily at a higher temperature. This saccharate is not very stable.

Barium forms one saccharate—the monobasic. This is not used in sugar manufacture owing to the scarcity of the reagent.

Lead forms three saccharates, of which, one, the tribasic, has been used to some extent in sugar manufacture.

Lead and barium are usually considered objectionable on account of their toxic properties.

While saccharates formed with each of the bases mentioned have been used, those of the greatest commercial importance are the tribasic saccharate of lime and the dibasic saccharate of strontium. The selection of the base depends largely on local conditions as to the ease of obtaining a supply of the material. The German factories use both lime and strontium processes, and the French and American factories, which cannot readily obtain strontia, use Steffen's tribasic saccharate-of-lime process, the separation modification.

In order that a clear idea of the development of these precipitation processes may be formed, methods that are no longer employed, as well as those in current use, will be described.

28. Elution Processes.—The original elution process, which is one of the oldest precipitation processes, was invented by Lair and Bilange, in France. In this process, the sugar is precipitated as a tribasic saccharate of lime. On the addition of powdered quicklime to the molasses, the saccharate immediately forms and the mass solidifies. The solid mass is broken into small fragments and is then extracted with water so as to remove the impurities. The saccharate either is used in liming the juice, preparatory to the carbonation, or is heated nearly to the boiling point in water and is carbonated and subsequently treated in the same manner as juices.

Scheibler, and later Seyferth, modified the elution process, using 37 to 40 per cent. alcohol for washing. They later recovered the alcohol by distilling.

There is, however, no factory now, either in this country or in Europe using the elution process. For this reason a further description of the process is unnecessary.

29. Steffen's Substitution Process.—If the monosaccharate of lime in water solution is heated to boiling, all the lime will combine with one-third of the sugar and form the tribasic saccharate, which is not readily soluble in water. The remaining two-thirds of the sugar is set free. These properties of the saccharates were utilized by Steffen, an Austrian engineer, in his *substitution* process, which is of importance as the forerunner of his *separation* process, now widely applied in the American beet-sugar factories.

The molasses is diluted to a sugar content of about 10 per cent., and then sufficient lime is added (28 per cent. of the sugar) to form a monosaccharate with the sugar. After agitating the mixture for 8 to 10 hours at a temperature of about 10° C., it is transferred to closed vessels, where it is heated under pressure to a temperature of 110° C. The tribasic saccharate of lime, which separates at this temperature, is removed by filtration through presses, and is then suspended in 120 per cent. of its weight of boiling water and again filter-pressed; the cake is washed with 35 per cent. of boiling water. The tribasic saccharate is utilized as has been previously described. To the mother liquor and the filtrates from the second filter pressing is added sufficient molasses to restore the sugar content to 10 per cent. of the solution. In other words, the sugar of a fresh portion of molasses is substituted for that removed in the tribasic saccharate; hence the name **substitution process**. This diluted molasses is treated as before, and one-third of the sugar it contains is removed in the saccharate. As a rule these manipulations are repeated about twenty times, fresh molasses equivalent to the sugar removed being substituted each time, until the mother liquor becomes so impure that it can be treated only with difficulty. At this stage of the process, the sugar, with the exception of a small quantity, is removed by repeated precipitations without the addition of molasses.

This process has been superseded by the Steffen separation process.

30. Steffen's Separation Process.—The Steffen separation process is used by many American beet-sugar factories

and yields very satisfactory results. This process depends on the precipitation of all the sugar in the molasses as the tribasic saccharate of lime, under temperature conditions that produce a very granular precipitate. The precipitant is freshly burned quicklime that has been reduced to an impalpable powder.

The process is conducted as follows: A quantity of molasses is reduced with water to a density of 10°–12° Brix, and to a percentage of sugar of about 5–7. The solution is placed in a closed mixer provided with cooling tubes and is cooled to as low a temperature as possible (10° C. to 20° C.). The weighed lime powder is then added in a continuous stream by a screw conveyer. At a low temperature, the lime combines with the sugar in preference to the water, and, therefore, does not hydrate. The lime addition is continued until a test sample shows that all the sugar has been precipitated. In making this test, it is customary to filter the test sample through paper and then to take the specific gravity of the filtrate. The Brix hydrometer is used for this purpose, and should read in the filtrate, from 5° to 6½° according to the density of the original solution, when the precipitation is completed or, preferably, until the density no longer changes with the addition of lime. In the early work with this process, from 130 to 140 pounds of lime was used to precipitate the sugar from 100 pounds of molasses, but this quantity has been reduced to about 50 pounds.

31. The tribasic saccharate produced at low temperatures under conditions described is very granular and may be readily separated from the mother liquor by filter pressing under low pressure. The crude saccharate suspended in the mother liquor resembles dirty milk of lime. The mother liquor is removed by filtration, and as it contains the potash salts of the molasses, it may be used as a fertilizer, or the potash may be recovered. The saccharate is washed in the filter press with cold water, and the wash water, which contains saccharate of lime in solution, is used in diluting a fresh lot of molasses.

32. The washed precipitate contains from 90–95 per cent. of the original sugar in form of a rather pure tribasic saccharate

of lime, and is used in liming the hot juice for the carbonation. The saccharate may be reduced to a thin milk with hot water and the lime precipitated with carbonic acid, leaving a very pure sugar solution. It is necessary to use very hot juice or hot water in reducing the saccharate to a milk, since in the cold, the carbonic acid causes the granules to become coated with carbonate of lime, which occludes particles of the undecomposed saccharate and results in a loss of sugar.

The Steffen separation process requires the installation of a refrigerating plant, except when used during the winter months in cold climates.

With a view to the economy of lime, Bauer devised and patented a continuous rotary kiln for calcining the lime cake from the carbonation. The lime produced is again utilized in the saccharate process. This type of kiln is used in several American factories.

STRONTIUM PROCESSES

33. Two methods have been devised by the eminent sugar chemist, Doctor Scheibler. These are known as the *strontium dibasic* and *strontium monobasic processes*. The dibasic process was patented in 1880 and the monobasic in 1882. The latter never has been a success and is not used anywhere at present.

34. Dibasic Strontium Process.—The hydrate of strontium, used as a precipitant in the dibasic strontium process, is obtained from the mineral strontianite—carbonate of strontium. The strontianite is calcined, and the resulting oxide is converted into hydrate by treatment with hot water. The hydrate dissolves, and the solution is separated from the impurities by decantation and crystallization. After the factory is once supplied with strontium hydrate, only a small quantity of strontianite has to be calcined from time to time to compensate for losses in the manufacture.

35. The hydrate of strontium in solution and the molasses are heated to the boiling point and mixed together in a tank

provided with a steam coil or a blow-up pipe. The strontium is added in excess of the quantity required to produce the dibasic saccharate. The mixture is boiled, and saccharate separates in a granular form. The saccharate and the mother liquor are thrown on a metallic screen, and the liquor is drawn off by suction. The precipitate is washed with a hot 10-per-cent. solution of strontium hydrate, and the washings are used in preparing another quantity of the saccharate. The mother liquor is cooled, and the excess of the hydrate is recovered from it by crystallization. The saccharate is transferred to a series of communicating tanks, and water cooled to below 15° C. and containing 2 per cent. of strontium hydrate is passed through the saccharate in the first tank, thence to the second tank, and so on. During this operation, cold water circulates about the tanks to cool their contents. At this stage of the process, the saccharate decomposes, the sugar and a part of the strontium passing into solution and the strontium hydrate crystallizing. The hydrate so formed is used in treating a fresh quantity of molasses. The sugar solution is carbonated, and the strontium carbonate is separated in filter presses and recalcined and treated as described for the strontianite. In lieu of the separation of a part of the strontium hydrate from the sugar by crystallization, it may all be precipitated by carbonic acid. The sugar solution is utilized in the same way as at other stages of the manufacture.

MISCELLANEOUS MOLASSES PROCESSES

36. Many modifications of the molasses processes described have been devised and abandoned; also, other processes have had a very limited application. The latter processes include precipitation of the sugar as barium saccharate and as lead saccharate.

37. Remarks on the Molasses Processes.—The osmose and the Steffen separation are the only processes used in America. Owing to the abundant deposits of strontianite in Germany, the strontium process is extensively employed in that country, but it is seldom used elsewhere.

UTILIZATION OF BY-PRODUCTS

38. Exhausted Cossettes, or Pulp.—In Europe as well as in the American factories the by-product of beet-sugar manufacture, namely, the exhausted cossettes, or pulp, is fed to cattle and sheep. For this purpose, the pulp is conveyed from the diffusion battery to pulp silos or to continuous presses of the Klusemann, Bergreen, or other type, and a part of the water it contains is removed from it.

39. The *Klusemann press* is a device for forcing pulp through a perforated iron cylinder, in which is placed a cone-shaped shaft that is fitted with steel blades arranged in the form of a helix. The conical shaft is also perforated. The cylinder has a jacket that leads the water pressed from the pulp to outlet pipes at the bottom; a pipe also conducts the water from the inside of the cone. As the pulp descends in the cylinder, the space it may occupy becomes smaller and smaller and the consequent pressure forces the water from it. The wet pulp is fed continuously into the cylinder at the top, and the pressed pulp is discharged at the bottom. The wet pulp contains about 95 per cent. of water and the pressed pulp, 87 per cent. The efficiency of the pressing depends somewhat on the quality of the beets and the working conditions in the diffusion battery. Thin cossettes from sound beets that are extracted without excessive heat and delivered to the presses at a temperature of about 60° C., are readily pressed.

40. The wet cossettes are often stored in large piles on the ground near the factory, drainage ditches being provided to carry off the water. The upper surface rots, but the lower layers keep fairly well and are greedily eaten by cattle. This method of storing, however, results in a great loss of nutritive matter. The pulp is often stored in cemented excavations, or silos, that have a covering of straw and earth. In such cases, arrangements must also be made for the removal of the drainage water. In recent years, much attention has been given to the drying of beet pulps for shipment. The dry pulp is used as cattle food and as an absorbent for a liquid food,

such as molasses or blood or a mixture of these. There are several kilns in use for drying the cossettes, of which the Buettner and Meyer dryer in its latest modifications is about the most perfected and efficient one. The temperature of the kiln may be high, but owing to the rapid evaporation, that of the pulp does not reach 100° C. The dried pulp contains from 12 to 15 per cent. of moisture. It is not necessary to reduce the water content below these figures, since the dried pulp will immediately reabsorb this quantity on exposure to the air. The dried-pulp or molasses-pulp foods prepared in this manner may be shipped in bags, and under proper storage conditions will keep indefinitely.

41. Molasses.—In factories that do not use a saccharate process, there is a large molasses residue. This is utilized either by mixing it with dried beet pulp for use as cattle food or by fermenting and distilling it, with the subsequent recovery of the potash salts from the final residue, which is termed *vinasse*.

The molasses is prepared for distillation by dilution and fermentation. A very small proportion of sulphuric acid is often added to the diluted beet molasses in order to promote the vinous fermentation and to check objectionable ferments. After distilling off the alcohol from the fermented molasses, the *vinasse* is evaporated to a thick liquor in multiple-effect evaporators or in large iron pans and the residue is then ignited. The organic matter burns off, leaving a black ash that is very rich in potash salts. The salts are recovered by lixiviation, followed by concentration and crystallization.

42. In this process, as usually applied, all the nitrogen is lost. It has been proposed by a French chemist, M. Vasseux, to concentrate the *vinasse* to a certain density determined by experience and then to add sufficient sulphuric acid to it to convert practically all the potash salts present into a sulphate. Under suitable conditions of concentration, nearly all the potash sulphate crystallizes and it is removed by means of a centrifugal. The mother liquor is concentrated and used as a nitrogenous fertilizer. The nitrogen may also be saved by submitting the dried *vinasse* to dry distillation in a retort.

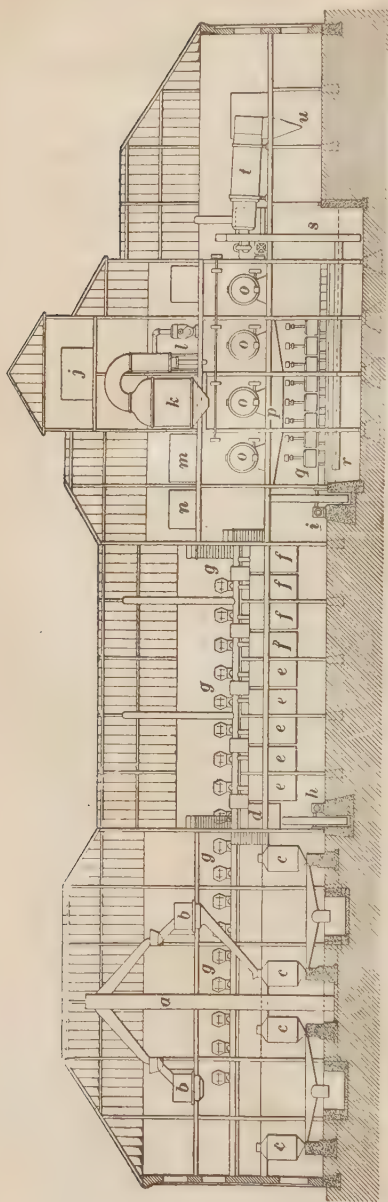


FIG. 5

The molasses from the beet is not usually suitable as food for human beings. Both its taste and its odor are very disagreeable. Several processes have been devised, notably in Germany, for removing or disguising the disagreeable properties of the molasses so as to make it fit for table use.

ARRANGEMENT OF MACHINERY IN A BEET-SUGAR FACTORY

43. Fig. 5 illustrates a section of the Los Alamitos sugar factory, erected in 1897 by E. H. Dyer and Company. Referring to this illustration, *a* shows the beet elevator; *b*, the beet cutter from which the cossettes are delivered into the diffusion battery *c*; *d*, a receiving tank; *e*, the first carbonators; *f*, the second carbonators; *g*, the filter presses; *h*, the engine that drives the beet slicers and elevators; *i*, the engine that drives the centrifugals and crystallizers; *j*, the water tank, to supply the diffusion battery; *k*, the strike, or

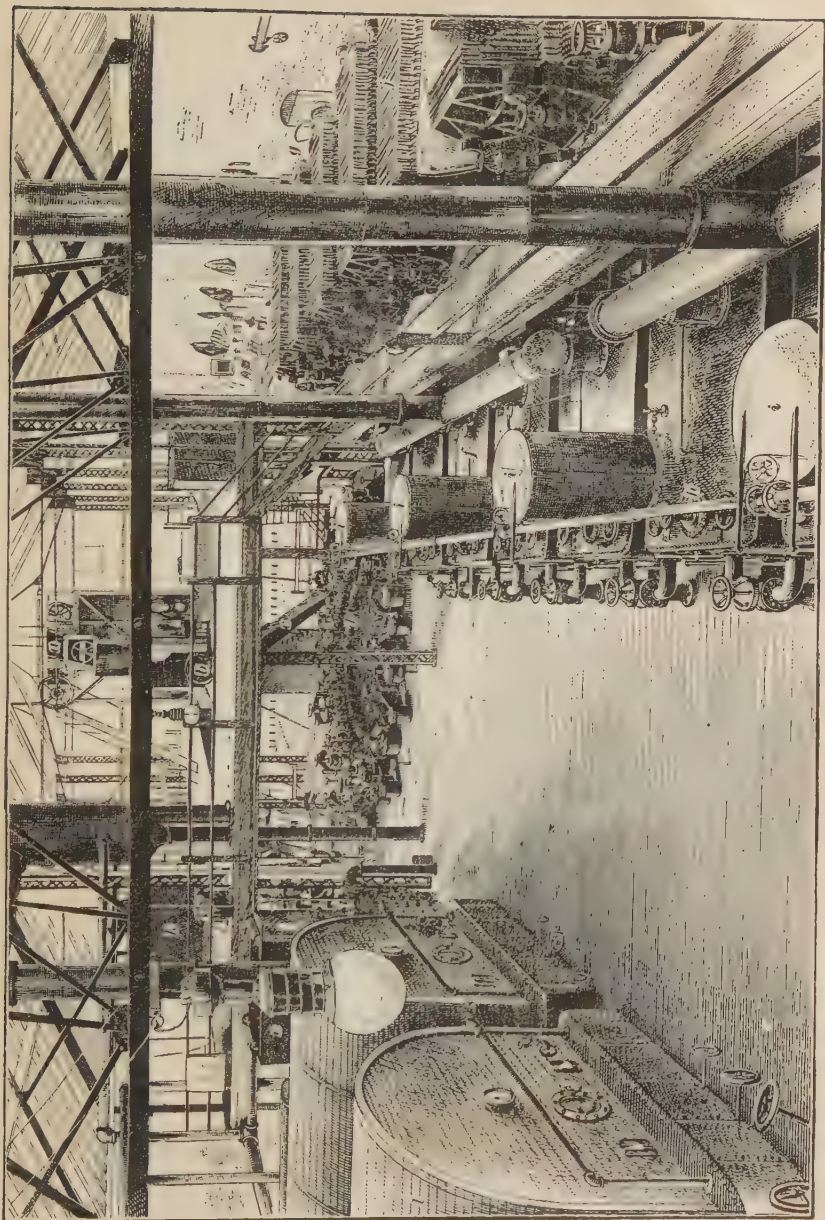
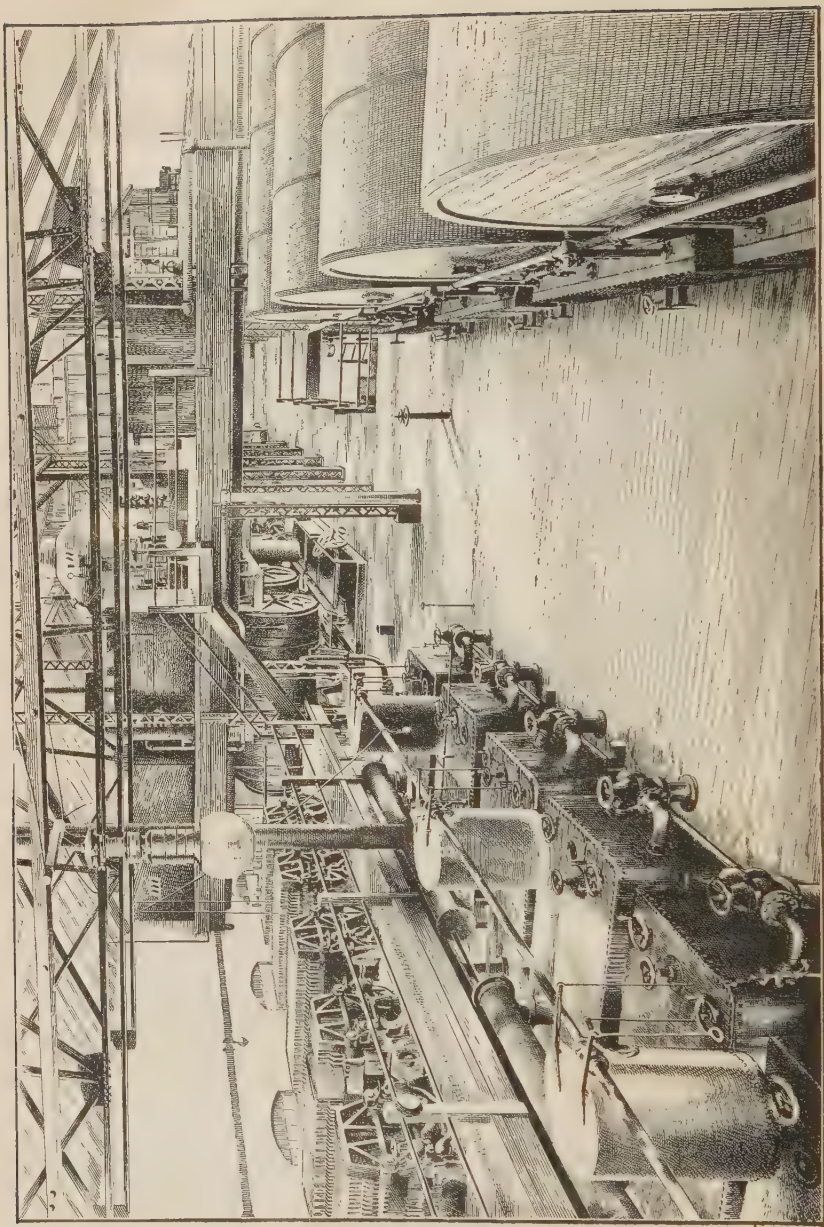


FIG. 6

FIG. 7



vacuum, pan; *l*, the air pump, to remove air and gases from the strike pan; *m*, the tank, to hold the sirup preparatory to being boiled in the strike pan; *n*, the molasses tank; *o*, the crystallizers; *p*, the mixer; *q*, the centrifugals; *r*, the conveyer that carries the sugar from the centrifugals and delivers it into the elevator *s*; *t*, the granulator, or sugar drier; and *u*, the hopper, from which the sugar is packed for shipment.

44. Figs. 6 and 7 are illustrations showing the main floor of a sugar house. At *a*, Fig. 6, is shown the elevator that conveys the beets from the washer to the slicer *b*, where they are cut into cossettes, and at *c*, the chute through which the diffusers of the battery *d* are charged with cossettes. The exhausted cossettes are dropped into sluices, or conveyers, below the battery, where they are loaded into wagons, and then taken to the silos, or storage place, to serve as food for cattle. The diffusion juice is measured in the tanks *e* and then flows into the carbonation tanks, or carbonators, *f*. After the liming, using milk of lime from the measuring tanks *i*, and the carbonation, the juice is filtered through the presses *g*, and the filtrate is conveyed to the saturation, or second-carbonation, tanks, *h*. When the saturation is completed, the juice is again filter-pressed, this time in the presses *j*, and the filtrate is pumped to the sulphuring tanks *k*. After sulphuring, the juice is filtered through mechanical filters, not shown in the figure, and the filtrate is concentrated in the quadruple-effect evaporator *l*. The sirup from the evaporator is sulphured and passed through mechanical filters, not shown, and is stored in the charge tank *m*, Fig. 7, ready to be boiled to grain in the vacuum pan *n*. The massecuite is conveyed from the vacuum pan to the mixer *o* of the centrifugal machines in order to be purged, or first to the crystallizers *p*, according to the method of manufacture.

REFINING OF SUGAR

INTRODUCTION

45. The refining of raw sugar is conducted on a very large scale in many of the seaboard cities of the United States. With few exceptions, the factories of Louisiana and Texas produce raw sugars, though at one time almost the entire product of these states was suitable for immediate consumption. From the introduction of the industry, the American beet-sugar factories have, with only two or three exceptions, manufactured a high grade of granulated sugar, ready for consumption. As a rule, the tropical countries produce sugar suitable only for refining.

46. Many grades of raw sugars are imported for the American refineries, certain establishments preferring and working certain grades for which they are best equipped. Table I, in which are given the analyses of a few typical grades of raw sugars, has been prepared from data obtained by Dr. W. D. Horne, chemist of the National Sugar Refining Company and supplied through his courtesy.

TABLE I
ANALYSES OF RAW SUGAR

Country	Grade	Per Cent. Sucrose (Polarization)	Per Cent. Ash	Per Cent. Water	Per Cent. Glucose	Per Cent. Organic (Non-Sugar)
Cuba.....	Centrifugals...	95.30	0.66	1.55	1.10	1.39
Cuba.....	Muscovado....	88.53	1.20	4.43	3.05	2.79
Cuba.....	Molasses sugar.	88.25	1.66	3.88	2.70	3.51
Java.....	Centrifugals...	97.60	0.41	0.53	0.82	0.64
Mexico.....	Centrifugals...	94.67	0.66	1.48	1.70	1.49
Mexico.....	Muscovado....	89.39	1.33	3.50	2.47	3.31
Mexico.....	Molasses.....	89.32	1.43	3.45	2.64	3.16
Macoris.....	Concrete.....	83.15	2.42	4.52	3.68	6.23
Macoris.....	Centrifugals...	96.27	0.48	1.05	0.99	1.21
Macoris.....	Muscovado....	92.20	0.86	3.75	1.61	1.58
Macoris.....	Molasses sugar.	86.71	2.53	4.08	1.86	4.82
Germany.....	Beet sugar.....	94.57	1.01	2.26	2.16
Austria.....	Beet sugar.....	94.74	1.12	1.80	2.34
Hungary.....	Beet sugar.....	94.57	1.05	2.21	2.17

47. This list given in the table does not include some of the very low-grade sugars. The grade known in the market as *centrifugal* is produced from sugar cane and is of the first crystallization in a vacuum pan. The modern raw cane-sugar factory produces little except the centrifugal sugar. The *muscovado* is made from cane, but the sugar is grained in coolers from liquor that is concentrated in an open pan, or kettle. *Molasses sugar* is made from cane molasses and is obtained from strikes boiled *blank* or to *string-proof*. A similar sugar, called *beet seconds*, is made from beet molasses. The grade known as *concrete* is obtained by concentrating cane juice until it solidifies on cooling. The evaporation is usually conducted in open vessels. Concrete, therefore, contains all the impurities of the clarified juice. The sugar that is usually called *beets* by the refiners is the first crystallization of beet sirup.

The centrifugal sugar is the best refining grade. This is followed by beets and muscovado. Molasses sugar, on account of its soft grain and high percentage of impurities, grades below the muscovado. The expense of refining concrete sugar is very high, since the impurities usually eliminated in the factory are brought with it to the refinery.

48. Raw cane sugars are sold to the refineries on a basis of their polarization and grade. There is an arbitrary deduction made from the polarization of beet sugar to determine the *titrage*, or *test*, on which the price is based. This deduction is generally five times the percentage of ash in the sugar. On the importation of raw sugar, the duty is based on the polarization of the sugar, together with its color, as referred to a set of colors prepared in Holland and called the *Dutch standards*.

Ninety-six degrees, polariscopic test, is the usual standard for centrifugal sugar. A deduction is made for each degree that the test falls below 96° and additional sum is paid per degree above this number, namely, $\frac{1}{16}$ cent per degree above and $\frac{1}{8}$ cent per degree below 96°. This scale is made on the assumption that sugars below the 96° test are more difficult to refine

than those of 96° or higher. Muscovado and molasses sugars are sold on a basis of 89° test.

As already indicated, the method of refining depends largely on the grade of the raw sugar. The process used for the highest refining grade, centrifugal sugar, will now be considered.

SUGAR-REFINING PROCESS

49. The centrifugal sugar is dumped into an elevating mixer, called a *magma machine*, where it is mixed with sirup so as to form a magma of about the consistency of massecuite. As fast as the magma is formed, it is elevated and discharged into the mixer of the centrifugal machines. The sirup softens the coating of impurities that is on the crystals and prepares the latter for washing in the centrifugals. As is customary with massecuites, the magma is purged in the centrifugals, and the crystals are thoroughly washed with water. The resulting sugar is of a very light straw color and of a polariscopic test about 99°. This sugar is next dissolved in the melters, forming a solution containing about 50 per cent. or more of sugar, and is then pumped to steam blow-up tanks. The sirup obtained in washing the raw sugar is pumped to separate blow-ups for treatment apart from the melted sugar, since the sirup contains most of the impurities of the raw material. This method enables the refiner to work 89° test beet sugar with cane sugar. The washed sugars are mixed, and the wash sirup containing most of the impurities is mixed with other low-grade materials.

The melted sugar is limed to slight alkalinity in the blow-up tanks, and then sufficient phosphoric acid is added to combine with the lime. After thoroughly blowing up the liquor with steam and skimming it, it is filtered through bag filters.

50. Taylor Filters.—The filtering apparatus used in sugar refining is known as the 'Taylor filter. The bags of this filter are about 5 or 6 feet long and from 4 to 6 inches in diameter. These bags are made of strong, twilled cotton

cloth, and each is slipped into a slightly longer outer bag, or sheath, made of strong coarse cloth. The mouth of each filtering element is tied over a nozzle, which is then screwed into a suitable hole in an iron plate. This plate forms the bottom of a very shallow tank and the top of the filter box, or chamber. The filter box is usually of a size sufficient to accommodate a large number of filter bags, and is provided with suitable doors to give access to the interior; it also has steam connections and pipes for filtered liquor.

51. During filtration, a very shallow depth of liquor is maintained in the feed-tank. The bags gradually fill with the lime precipitate and the impurities that are separated during the treatment of the liquor in the blow-up. At this stage, the mud in the filter bag is washed with hot water, while steam is admitted into the filter box to maintain the temperature. The wash water is utilized in melting sugar or in some other process, so that none of the sugar it contains will be lost. The mud is washed from the bags and then filter-pressed. The filtrate and the water from washing the bags are concentrated in order to recover the sugar they contain. It should be noted that in the case of the Taylor, or bag, filter the filtration takes place inside of the bag. This is just opposite to the way that filtration is carried on in mechanical filters used in sugar factories.

The sirup molasses drained from the raw-sugar crystals in purging the magma, as previously described, is also treated the same as the melted sugar, but on account of its low purity, it is kept apart from the latter at all except the last stages of the process.

52. Char Filters.—From the bag filters, the liquor is conveyed to the char filters, which are large, vertical iron cylinders with perforated false bottoms. There is also a tight-fitting door, or cover, at the top of the filter and a discharge door for char at the bottom. Pipes and valves are arranged to conduct the liquor into any filter and to draw off the filtrate. The filtration is conducted under pressure, and the discharge pipes for the filtered solutions terminate above the filters in

plain view of the attendant. The filtering medium is animal charcoal prepared from bones. This material, in small fragments, is packed on blankets, which are laid on the false bottoms of the filters. The strained sugar solution from the bag filters is run into the char filter until it displaces the air and completely fills the filter. After a certain length of time, determined by experience, the discharge and inlet valve are opened and a continuous stream of sirup, or final molasses by-product, is passed through the filter from top to bottom. The pipes discharge into a conveniently arranged trough near the top of the filter. For some time the liquor flowing from the filter is water white and clear; it then has a faint tinge of yellow, and, finally, when the color deepens, water for washing the char is substituted for the liquor. The washing is continued until tests show that the water contains too little sugar to justify saving it. The wash waters are concentrated to heavy liquors in multiple-effect evaporators. The sirup is graded according to its color.

53. During the filtration, the char absorbs certain organic and inorganic impurities and a small quantity of sugar from the sirup and decolorizes the latter. The filtered liquors are boiled in vacuum pans for the crystallization of the sugar, as in the factory processes previously described. The molasses from the sugars is boiled in just as long as its color will permit without affecting the quality of the refined sugar, or it is used in making soft, yellow sugars. The sirup obtained in the first stages of the process of refining is treated precisely the same as the liquor from melted sugar, and when the molasses from this sirup becomes too highly colored, it is mixed with the same grade from the melted sugar, to produce raw sugar. The very low grades are finally treated in the crystallizers, as in the factory methods.

54. Molasses sugars are often mixed with centrifugal sugar at the factories and also at the refineries, and are then treated as described for centrifugal sugar. Certain very low-grade sugars are melted, and the resulting liquors are worked separately from the high-grade sugars. The sugars are purged in

the centrifugals and are washed and dried, or granulated, as in the factory methods.

The product known as **cube sugar** is made by moistening soft, white sugar with white sirup and molding it in a special machine. The cubes are dried in an oven. **Loaf sugar** is cut or partly sawed and broken from molded slabs, or loaves. **Powdered sugar** is produced by grinding or crushing the crystals.

The factories of continental Europe use sulphurous acid to some extent for bleaching the liquors, so as to economize bone black, and depend on systematic washing of the sugar with sirup to remove the molasses.

55. Revivification of the Char.—The bone black, or char, used in decolorizing the liquors, soon ceases to be effective through the absorption of organic and mineral matters. The char is not rejected, however, but is revived by washing and reburning.

The washing is effected in the filter by passing boiling water through the char. In European practice, where raw beet sugar is almost exclusively used for refining, the char is sometimes washed with dilute hydrochloric acid also, so as to remove the carbonate of lime. After thorough washing with water, the char is conveyed from the filters to the kilns for reburning. The conveyer deposits the char on top of the kiln for a preliminary drying.

56. The char kiln used by refineries usually consists of a number of small, vertical, cast-iron pipes, about which circulate the heated gases from a furnace. The upper ends of the pipes terminate in a storage or drying platform for the wet char, and the lower ends, in sheet-iron cooling pipes. The maximum temperature maintained in the tubes is about 650° C. The apparatus is so arranged that air will not enter the hot zone of pipes of the kiln.

The wet or partly dried char enters the upper parts of the pipes and gradually works its way downwards to the cooling pipes as charges are withdrawn. These pipes are provided with valves, or gates. As the char descends, it passes

through the highly heated zone of the pipes and the organic matter is carbonized; from the hot zone, it passes into the cooling pipes and is drawn off for use from time to time.

The color of the revived char is indicative of the efficacy of the treatment. A reddish char is underburned; a gray char shows that air has leaked into the pipes and part of the carbon has been consumed; a white char is overburned; a brilliant black char indicates proper burning.

57. On account of the absorption of mineral matter, the weight of the bone black increases with use. According to Gallois and Dupont, bone black of good quality that has been used should not weigh more than 1.23 times its weight when new. A gain of 47 per cent. in weight indicates that its condition is bad, and one of 50 per cent. that it should be rejected.

Refiners use a part of the spent bone black in manufacturing phosphoric acid, or superphosphate of lime, which is used for precipitating the lime when blowing up the liquors, as already described. The excess of spent bone black is utilized in the manufacture of phosphatic fertilizers.

58. By-Products in Refining of Sugar.—The only by-products in the refining of sugars is the *final molasses*, or *barrel sirup*. This material is often treated for the improvement of its color and is then utilized in the production of table sirup. It often reaches the hands of dealers that use it for mixing with starch sirup or glucose for table and bakers' use. Large quantities of the molasses are utilized in the manufacture of alcohol.



